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AFML ltr dtd 21 May 1973

AFML-TR-70-279

AD881276

EXPLORATORY DEVELOPMENT
OF IMPROVED OPTICAL FIBER BUNDLES

H. B. Cole
E. C. Hopkins
W. P. Siegmund

American Optical Corporation

Technical Report AFML-TR-70-279

January 1971

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Air Force Materials Laboratory
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio 45433

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FOREWORD

This report was prepared by the American Optical Corporation under Contract F33615-69-C-1391. This contract was initiated under Project 7320, "Fibrous Materials for Deceleration and Structures", Task 732001, "Organic and Inorganic Fibers". The work was administered under the direction of the Nonmetallic Materials Division, Air Force Materials Laboratory, Air Force Systems Command, with Walter H. Gloor, LNF, as Project Engineer.

This report covers work from March 1969 through June 1970. The manuscript was released by the authors in November 1970 for publication as a technical report.

This report has been reviewed and is approved.

J. H. Ross

J. H. ROSS
Chief, Fibrous Materials Branch
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ABSTRACT

The purpose of this contract was to conduct exploratory development on materials and techniques to improve glass optical fibers and fiber bundles with particular reference to coherent multifiber fiberscopes. Studies were carried out on improved interface formation, end tip fusing, experimental fiber drawing techniques and evaluation of various component glass, clad rods and fibers. Two basically different approaches to improving the core-cladding interface were explored: 1) the use of low melting fluxes with the rod and tube method, and 2) the use of a double crucible to completely melt the core and cladding glasses before drawing. Neither approach provided significantly better interface quality in the resulting clad rods.

A method was developed for fusing the end tips of coherent fiberscope bundles. Good results were achieved in bundle up to 3x3mm in cross section but considerable difficulty was encountered when the bundle size was increased to 10x10mm.

A number of different glass combinations were selected and drawn into clad rods and fibers in a search for improved transmission efficiency. It was concluded that the properties of commercially available glasses presently limit the fiber transmission and that glasses with improved bulk transmittance must be formulated if improved fibers are to be realized.

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1.0 Introduction

This final report summarizes work done under Contract F33615-69-C-1391 and covers the period from 1 March 1969 through 30 June 1970. The purpose of this contract was to conduct exploratory development on materials and techniques leading to improved glass optical fibers and fiber assemblies, with particular emphasis on the needs of coherent multifiber fiberscopes. The investigations were carried out in four general areas--interface studies, tip fusing, experimental drawing, and testing.

The interface studies were primarily aimed at obtaining a better core-cladding interface. Two approaches for making clad rods were tried that differed substantially from the normal rod and tube method. These were melt cladding and the use of fluxes by various techniques. In addition, several lesser variations from the standard drawing procedures were tested. The best results were obtained with a furnace design that represented a relatively minor change from the procedures that had been established over the years.

The tip fusing studies were directed at learning and solving the problems of fusing the ends of fiberscopes, starting with small sizes. Methods were found for avoiding blemishes, maintaining fiber alignment and overcoming the transition zone weakness for multifiber fiberscopes of 3x3mm format. The main problem of scaling up to larger sizes was determined to be in maintaining fiber alignment across the longer fused section required. Special techniques were developed for fusing of assemblies up to 10mm square with promising results.

The experimental drawing, although listed as a separate area of investigation, was largely to support the other areas. Many of the interface studies, for example, were primarily experiments in drawing. The tip fusing experiments required experimental ribbons for assembly and some experimental development was required to prepare ribbons with particular characteristics required for fusing. Much of the testing program was the evaluation of possible new glass combinations. Experimental drawing evaluated their fiber making characteristics and at the same time provided samples for optical test.

The testing effort was primarily to evaluate materials and experimental techniques. The attenuation constant of clad rods was found to be the most useful measurement, although a number of other measurements were made and new tests devised in order to answer specific questions.

2.0 Interface Formation

2.1 Background

The normal procedure for making multifiber fiberscopes involves two drawing steps. In the first step the core and cladding are joined and drawn into clad rods about 3mm in diameter. After inspection, some number of these (usually 36) are assembled for drawing into multifibers. In the second drawing step the individual elements are normally reduced to 10 microns, giving a 6x6 element multifiber 60 microns square. The multifibers are wound into ribbons and assembled to the desired format.

The core-cladding interface is formed in the first drawing step. In the second drawing step, clad-clad interfaces are formed, and the core-clad interface is reheated and stretched. The clad-clad interface can affect fiber quality, and the reheating of the core-clad interface can be significant, but in this project the main emphasis was placed on the formation of the core-clad interface during the first drawing step.

We normally use the rod and tube method for making clad rod. A rod of Schott F-2 flint glass is placed inside a tube of Kimble R-6 soda lime glass. Before assembly, one end of the tube is heat closed and both pieces are carefully washed, rinsed, and dried. Clean room procedures are used during assembly. A special stopper seals the other end of the tube and provides a connection to a vacuum pump during drawing. The pump reduces the pressure in the tube to 1/10 to 1/2 atmosphere. The reduced pressure helps in outgassing the surfaces as they heat but leaves some oxygen to burn any residual amount of combustible dust. The differential pressure collapses the tube, as it heats, and forces it against the core. This forms the core-cladding interface.

The clad rods are inspected by introducing intense light into the core and observing the side against a black background. Interface defects scatter strongly and even very small imperfections can be seen in this way. All rods have some imperfections. Bubbles appear to be the chief defect; very few glass combinations will show visible crystals at this stage. The bubbles appear to be the result of surface imperfections or foreign material on the assembled pieces. When dirt can be found, the scattering from the bubbles it causes is much greater than from the dirt itself.

The rod and tube method would appear to have definite limitations as to the quality that can be achieved due to the dependence on the surface quality of the components and the difficulty of completely controlling their history. The studies undertaken under this contract were aimed at eliminating the

dependence on surface quality by eliminating the exposed surface stage or by treating the surfaces in a way that would eliminate effects of their uncertain histories.

2.2 Direct Melt Cladding

The drawing of clad fibers from a melt was investigated as a way of avoiding the fusing of surfaces that have been exposed and possibly damaged or contaiminated. The work was done with a small platinum double crucible facility that had been used for small clad fibers and in which the orifices previously had been opened up in anticipation of drawing clad rod. This equipment was reconstituted and tested with the original single zone furnace using the standard F2-R6 combination. It was found that the flow of the two very dissimilar glasses could not be controlled adequately with the single heating zone.

A new furnace was built to provide separate temperature control of three heating zones along the length of the crucibles. A cross sectional view of the crucibles and this heating arrangement is shown in Figure 1 at approximately full size. This furnace gave sufficient control of the temperature pattern to permit the drawing of clad rods of normal dimensions using the lower portion of a standard clamp drawing machine to handle the rods. The quality of the rods was below standard, having many more than the normal number of interface bubbles. Figure 2 shows an end view of a rod cut through a large bubble.

The bubbles were identified as having originated on the wall of the inner crucible and carried into the interface when flow started. Two changes were made in the equipment in an effort to correct this problem. A furnace was built with higher temperature heaters for the upper part of the crucible. The crucible holding and adjusting mechanism was modified to permit replenishment during the run. Figures 3 and 4 show the arrangement used. Figure 3 is a photograph of the crucibles with the new support pieces, Figure 4 is a view of the heating assembly. The quartz-tungsten heaters gave a temperature capability of 1300°C versus 1000°C for the previous Kanthal heaters. This improved the fining or bubble removal from the glasses. Moreover, the replenishment capability allowed more of the starting bubbles to be carried away. The modifications gave better quality rods than obtained in previous double crucible tests, but even with a number of variations and refinements the melt cladding experiments were not successful in exceeding the quality of standard rods. In retrospect it appears that the experiments were simply on too small a scale for the size rod that was being made.

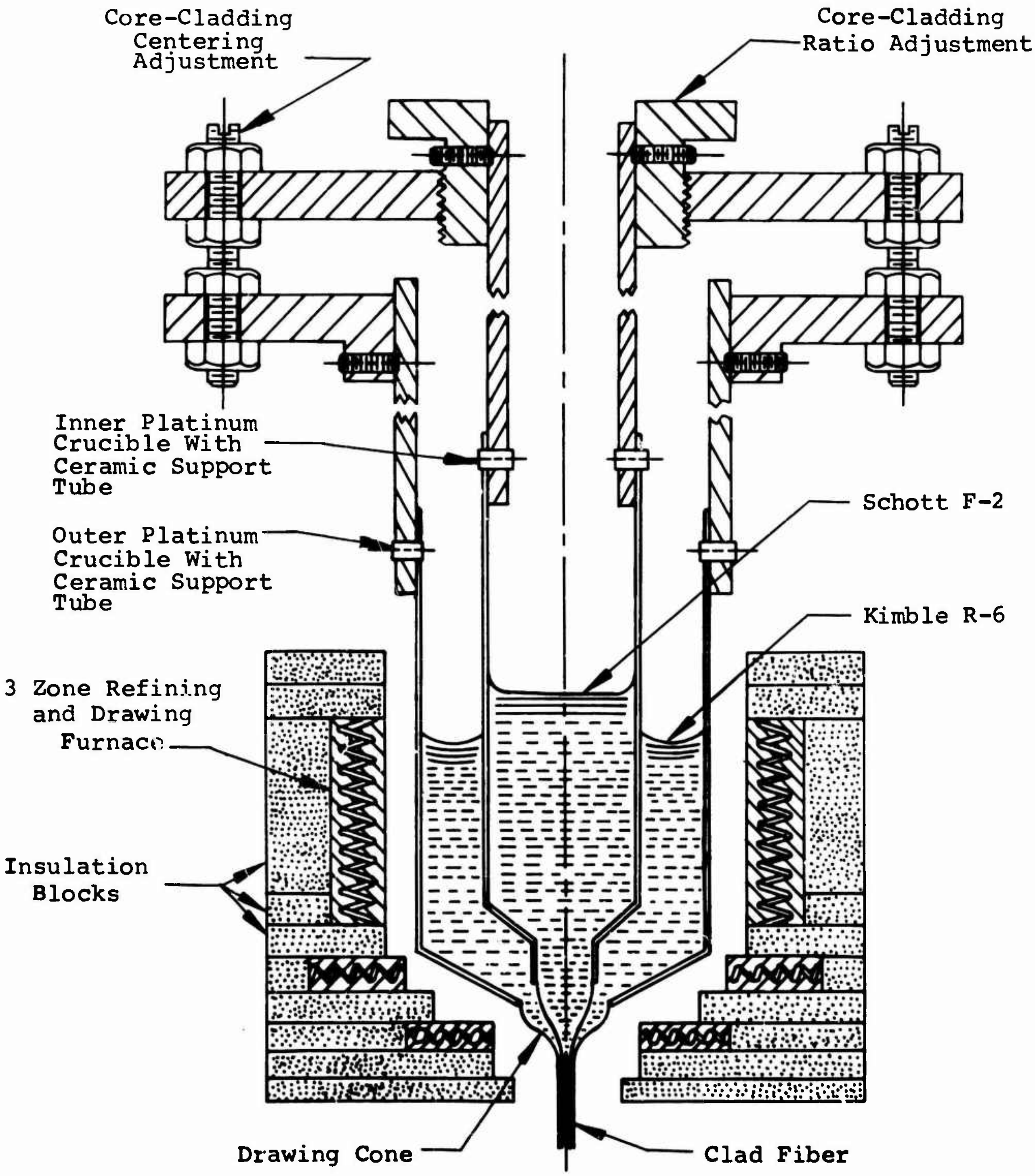

FIGURE 1

DIRECT-MELT FIBER DRAWING EQUIPMENT
DOUBLE CRUCIBLE-3 ZONE FURNACE

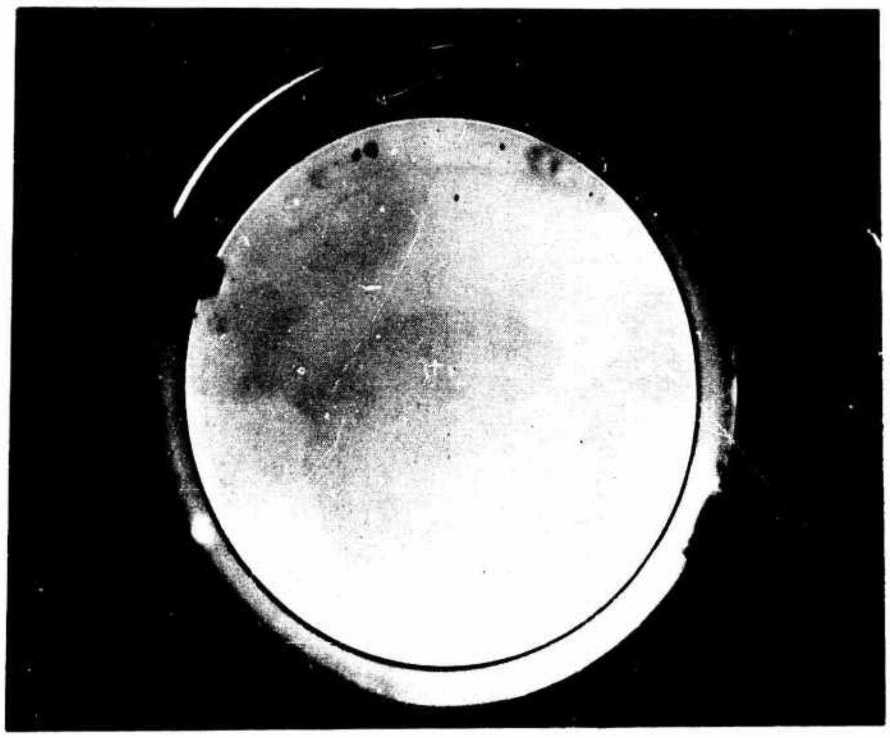

Figure 2 - Cross Section Through a 3mm Diameter Clad Rod Which was Drawn in the Double Crucible Showing Cladding Decentricity and a Large Bubble at the Interface

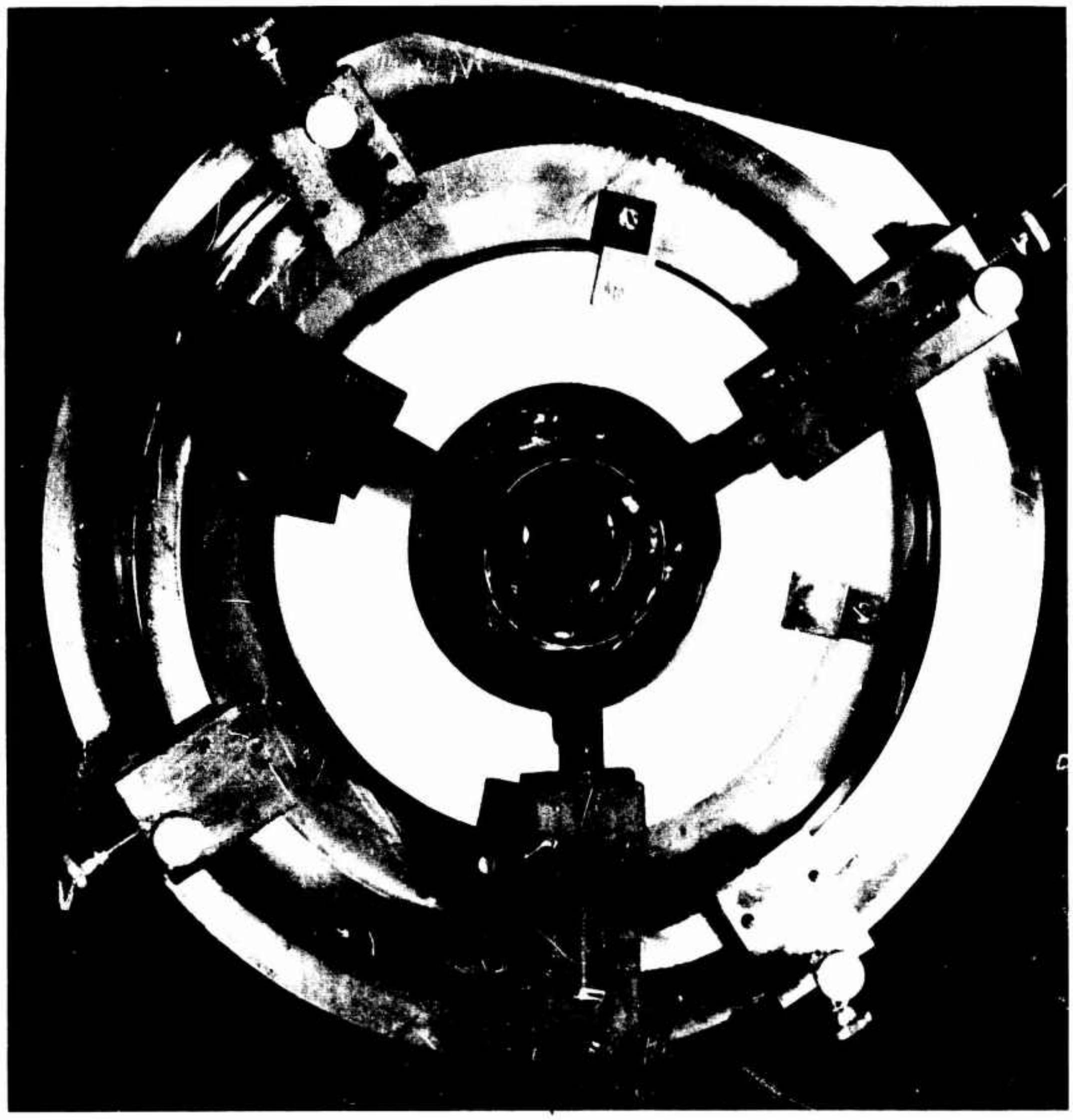

Figure 3 - Top View of Double Crucible and Support Structure

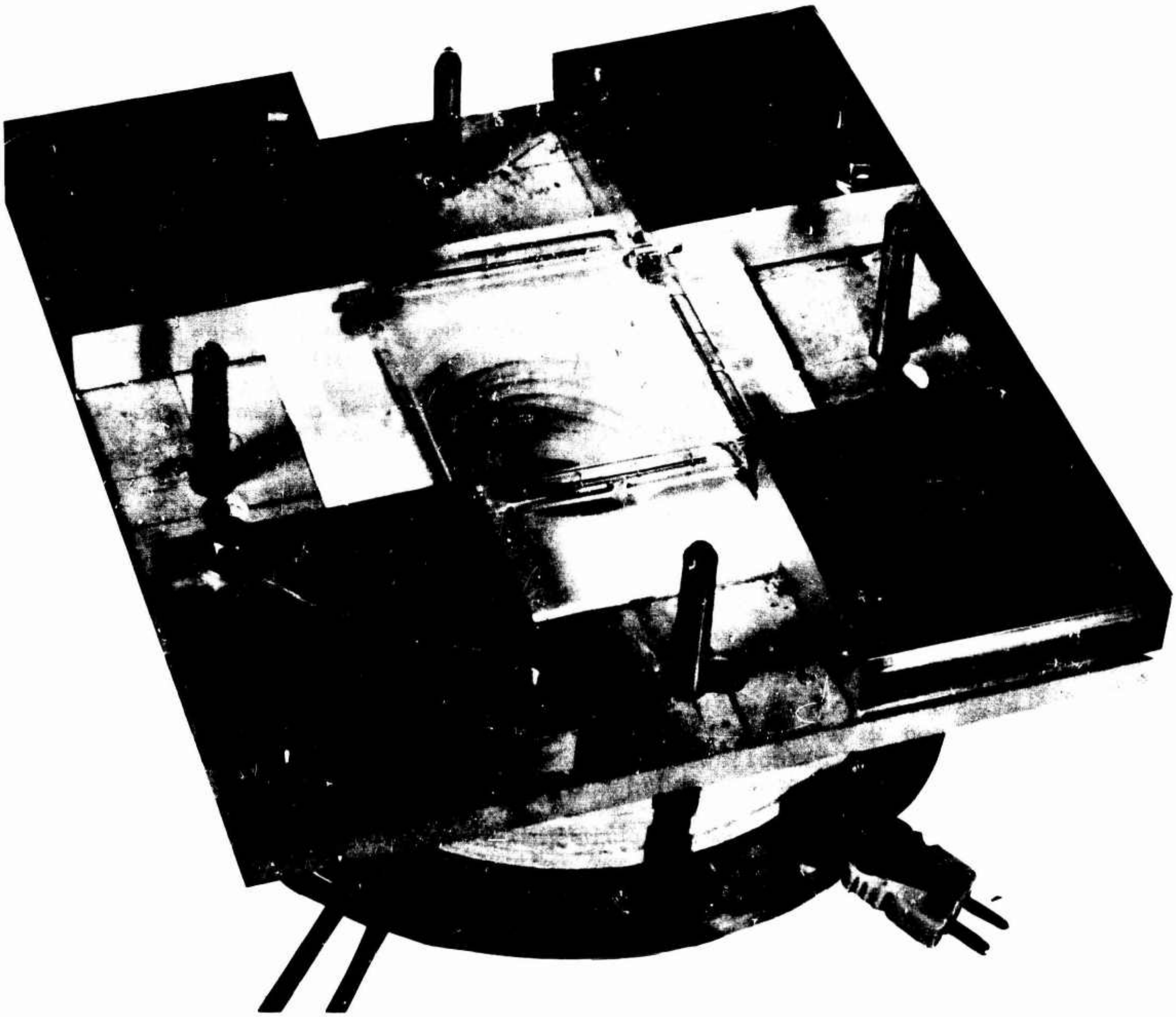

Figure 4 - Double Crucible Furnace

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2.3 Fluxed Interface Experiments

These studies were undertaken to determine the possible improvement in interface quality that might be obtained with the use of a flux. Fluxes have been found useful in the past in solving certain problems such as interface devitrification or lack of proper wetting between core and cladding glasses. The usual method of application has been as a frit of low melting glass, coating the core or the inside of the tube or both. This method of application, while helpful in many respects, does not appear to reduce the incidence of interface bubbles. Emphasis was therefore placed on the evaluation of other methods.

The first method evaluated was the use of the flux glass in the form of thin strips placed between the core rod and cladding tube. With this method of application even a small number of rods can coat the entire interface. The squeegee action as the tube collapses onto the core causes the softened ends of the strips to spread laterally until they fill the entire circumference. Used in this way the strips form a pool of flux. The pool grows during the first part of the run since the squeegee action allows very little of the flux into the interface. Eventually the pool becomes deep enough and the top of the pool becomes cool enough so that viscous forces cause flux to enter the interface at the same rate as new flux enters the pool. In general, the quality of the drawn rods appeared better before this equilibrium condition was reached.

The next mode of operation was with a fixed pool introduced at the beginning of the run. It was found that with a small pool a negligible quantity of flux would enter the interface and that the pool level would remain constant. The flux glass was introduced as a 3 to 5 gram block placed under the end of the core bar. The initial collapse of the tube onto the core forced the flux up into the space around the core.

This "flux pool" approach was used in most of the experimental work with fluxes. It was felt that defects on the core and inner tube surfaces would cause bubbles to form in the flux pool instead of in the core or cladding glass and that the trapping of a small amount of flux between core and cladding would be less deliterious than the trapping of a similar volume of gas.

In order to evaluate the flux pool concept adequately, it was necessary to find a flux material with the desired properties. The first requirement was low viscosity; it was considered desirable that the flux be very fluid at the temperature just above the drawing cone. The temperature is set by the viscosity of the cladding glass and the differential pressure used to cause it to collapse onto the core. Typically this is about 500°C. The next important property is resistance to devitrification at the required temperature. The fact that the flux does not move

through the furnace with the core and cladding glasses means that it is exposed to the temperature for a long period of time. This, in combination with the lower viscosity of the flux, makes the devitrification problem severe.

Low melting glasses were used in the initial flux experiments. These were mostly lead borate glasses of various compositions. The viscosities appeared to be marginally low enough. All had an excessive tendency to devitrify and send crystals into the interface.

Boric oxide was tried next. It did not devitrify, but was more viscous. It was concluded after several experiments that its viscosity was definitely too high for use with the flux pool technique.

Salts were considered next as possible fluxes. By selecting one with a melting point lower than the required operating temperature, the tendency to form crystals could be avoided completely and the viscosity was low because of the sharp transition from solid to liquid phases. Zinc chloride was tried first. Its liquid range from 262°C to 732°C, appeared appropriate, but there was excessive fuming because of the vacuum used. Silver chloride, M.P. 455°C, B.P. 1550°C, was tried next. It has the disadvantage of not being colorless, but its other properties seemed ideal.

A number of experiments were run with flux pools of silver chloride. In many respects the behavior of the bubbles in the pool was as desired. The bubbles formed at discrete sites on the core and tubing surfaces. The majority grew rapidly, rose to the surface, and broke. Many, however, clung to the core or tube surface and moved downward with it through the pool. The thickness of the pool, of course, decreased near the bottom as the tube approached the core. At some point the bubble was large enough to reach from the core to the tube. Once bubbles arrived at this point there was no tendency for the bubbles to break loose and rise to the surface. As the run progressed the number that were carried down this far increased and formed a collection in the lower part of the pool all around the core. When this happened the advantage of the pool was largely negated since gas was again carried into the interface. Figure 5 is a drawing of the flux pool set-up and shows the positions of the rod, tube, pool, furnace, etc. Figure 6 is a photograph of the flux pool during an experimental run and shows the bubbles adhering to the tubing wall.

These results indicate that the flux pool behaves only partially in the desired manner. The bubbles form in the flux as desired, but a stronger method is needed for removing them from the flux before they reach the bottom of the pool. Such a method may have to be fairly complex in view of the inaccessibility of the space involved. Two fairly simple approaches were tried and

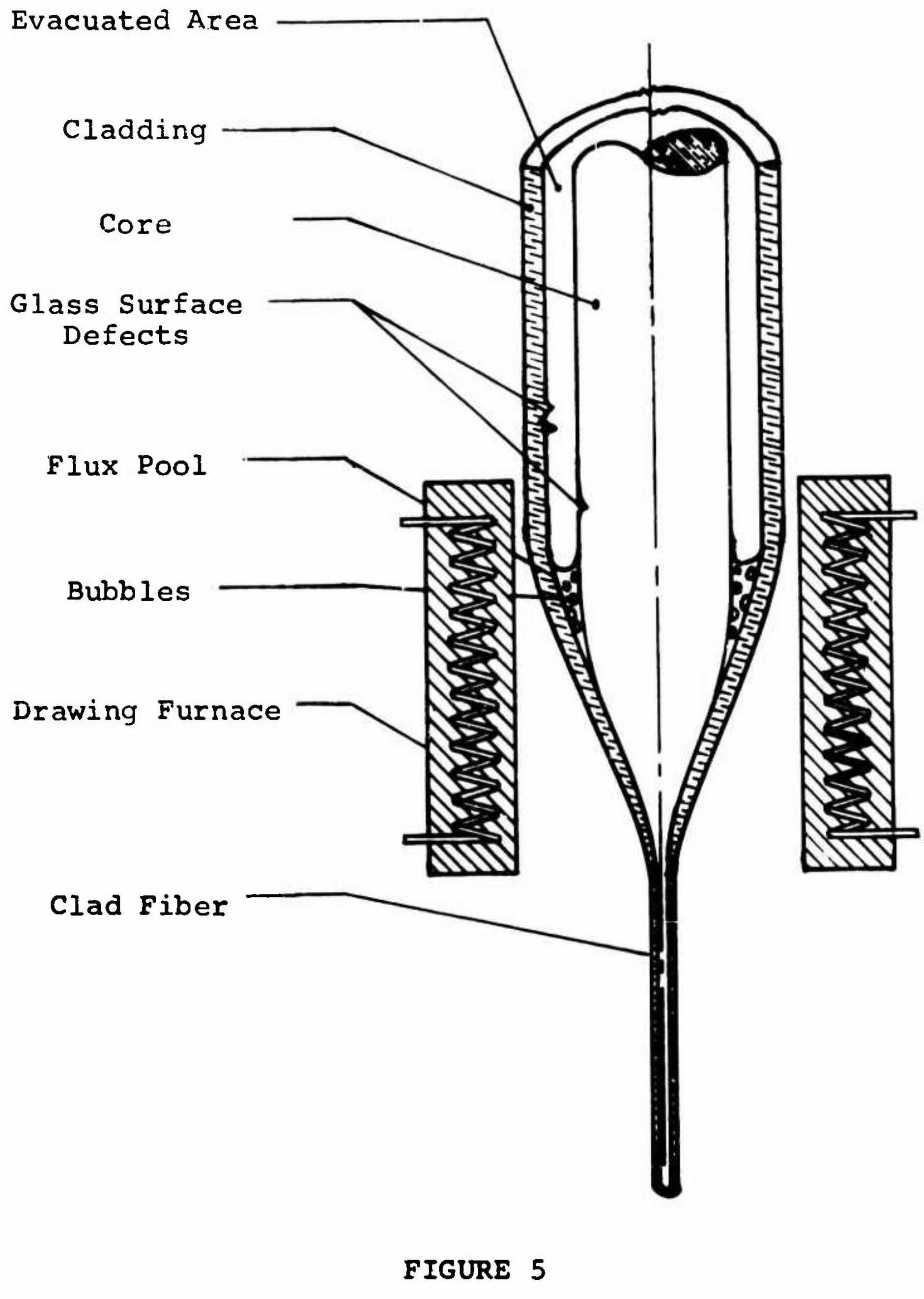

FIGURE 5

**REPRESENTATION OF "FLUX POOL"
FIBER DRAWING CONE**

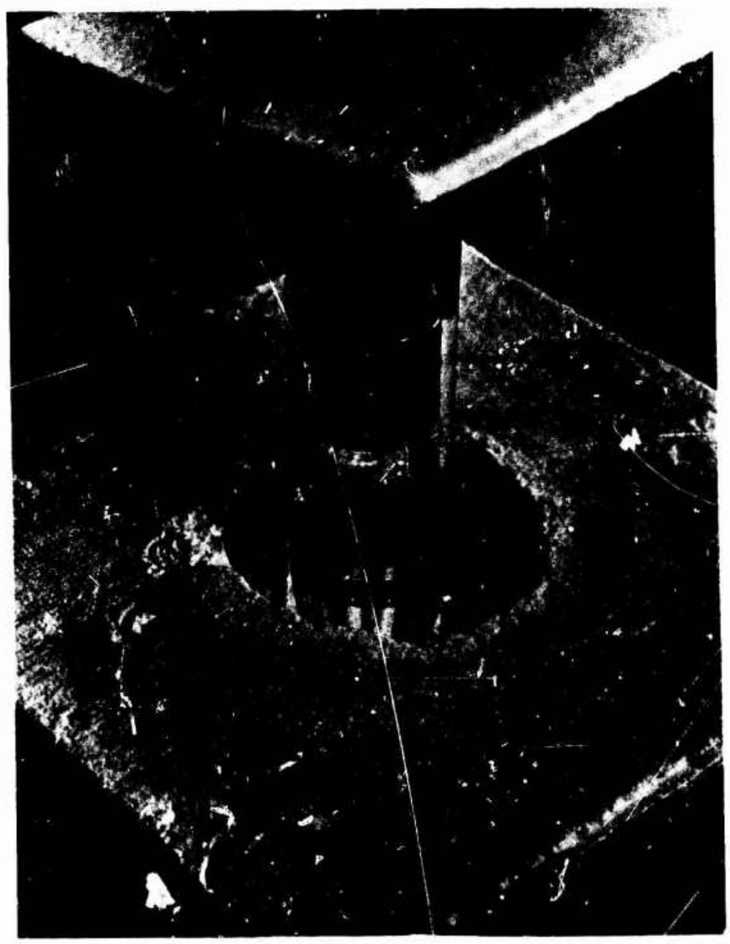

Figure 6 - View Into the Top of Drawing Furnace During the Silver Chloride "Flux Pool" Experiment. Bubbles can be Seen Adhering to the Inner Tube Wall.

found to be inadequate. One was the periodic cycling of the gas pressure above the pool from about .01 to .90 atmosphere every 10 to 15 seconds. This caused the bubbles to shrink and grow, but it did not greatly increase their tendency to break free from the surfaces to which they adhered.

A second possible method for causing the bubbles to break free was induction stirring. This was evaluated in a 5 KW induction heater using a pool of silver chloride in a standard test tube of borosilicate glass. The tube and flux were heated with a conventional radiant heater until the silver chloride melted. The test tube was then moved to the induction heater and the behavior of the pool and bubbles observed as the power was turned on and off. The liquid silver chloride was electrically conductive enough so that the temperature could be maintained with the RF field. The stirring action from the eddy currents, however, was not strong enough to greatly increase the tendency of the bubbles to break free from the tube wall.

It appears that for the flux pool to perform its desired function it would be necessary to provide a positive method of bubble removal. A mechanical wiper might provide the answer. The action would probably have to be strong enough to remove the nucleus that caused the bubble to start at that point. The other problem that would have to be solved is the selection of a colorless flux with the desired physical properties. The solution of these problems appeared to be beyond the scope of this project. Instead of pursuing this approach, effort was directed to the evaluation of more conventional process techniques that could affect interface quality. These are discussed in Section 2.4.

2.4 Other Interface Experiments

The measurement of the light attenuation per unit length in clad rods has been found to be a sensitive test for evaluating process variations. This test correlates well with the regular visual inspection of the rods and also with the performance of multifibers drawn from the rods. It was decided to take advantage of this test to evaluate process variations that would be expected to have less effect than those discussed in Sections 2.2 and 2.3.

The experiments tried included variations in glass preparation, variations in drawing procedures, treatment after drawing, and other procedures that differed considerably from normal methods. It was found that some relatively minor changes from standard methods had substantial adverse effects. The only change that appeared beneficial was a relatively minor change in the drawing furnace that possibly improves the zonal fusing action as the tubing collapses onto the core.

Two of the experiments had to do with glass preparation before drawing. Both gave adverse results. One involved the use of acid cleaning in place of the normal washing methods. The cleaning procedure for both the F-2 core and the R-6 tube consisted of a one minute acid dip followed by a one minute water rinse. The acid consisted of 12.5% HNO_3, 12.5% HF, and 75% H_2O. Drawing procedures were conventional except for the cleaning. The attenuation constant was .0030 per cm versus .0022 for good standard rods. The second method made use of a vacuum prebake intended to provide better outgassing of the surfaces prior to fusion than normally takes place as the glass feeds into the drawing furnace. In this experiment the core rod was placed in the cladding tube and, with the tube end open, placed in a vacuum furnace. It was baked in vacuum (about 1/1000 atmosphere) for nine hours at 425°C. It was then cooled under vacuum and immediately transferred to the drawing machine and drawn in the normal manner. The attenuation constant for the resulting rods was found to be .0040 per cm.

The results for the two experiments in surface preparation are consistent. Both were intended to remove the adsorbed water from the glass surfaces. The fact that both failed to produce improved results appears to indicate that adsorbed water is not a major problem or, at least, is removed in the normal heating at reduced pressure as the glass feeds into the drawing furnace. The substantial increase in attenuation in both cases is more difficult to understand. Possibly some slight crystallization is initiated by both the etching and the long bakeout.

Several of the experiments involved variations from the standard drawing method. The "normal" fibers are drawn in a furnace about 100mm long. The assembly consists of an F-2 core about 33mm in diameter in a tube 48mm in diameter. The pressure within the tube is reduced to 1/10 to 1/2 atmosphere so that atmospheric pressure outside the heated tube causes it to collapse onto the core. The assembly is drawn to a diameter of 3mm.

One experiment made use of a predrawing step for both the core and cladding in order to fire polish and outgas the surfaces prior to assembly. In this experiment, both components were drawn to 40% of their initial size before being assembled and drawn into 3mm clad rods. The results indicate that the reduction in assembly size and resulting change in required drawing ratio have undesirable effects that outweigh the advantages of having fresh fire polished surfaces to assemble. The attenuation constant for these rods was found to be .0034 per cm.

One experiment made use of a prefusing step in a high pressure fusing press to make a core-cladding assembly. An F-2 core rod was placed in a heavy R-6 cladding tube in a fusing die normally used for making fiber optic plate boules. Additional F-2 rods were added outside the tube to fill up the die. The assembly was fused in a normal fusing cycle. The assembly was

then removed from the die and the surplus glass ground off leaving an approximately uniform cladding layer. At this point the interface was inspected using immersion oil to permit viewing through the fine ground exterior surface. The interface was quite good except for some bubbles along the upper side, indicating that the fusion had been zonal from the bottom to the top but not along the length. One large bubble initiated a crack and caused a section of cladding to flake off. Figure 7 is a photograph of the fused assembly at this stage. (The core is illuminated to make interface defects visible). In spite of the flaked cladding it was possible to draw a number of 3mm clad rods from this assembly and to measure their transmittance. The measured attenuation constant was found to be .0034 per cm, about 50% worse than for the standard rods.

One experiment represented a substantial variation from normal drawing methods. It made use of an extra heating element in the annular space between the rod and tube during the mono drawing step. The purpose of the extra heater was to increase the preheating, outgassing, and fire polishing of the rod and tube surfaces before fusing together to form the core-clad interface. Because of the limited space the heater consisted of a stainless steel ring induction heated by means of an RF coil outside the cladding tube. The ring was supported by rods passing through the vacuum seal at the upper end of the cladding tube. Several variations were tried with the ring at different heights relative to the regular external drawing furnace and at different temperatures. Also, various amounts of insulation were tried around the ring in order to control the relative amounts of heat going to the rod and tube. The best results were obtained with the outside of the ring quite well insulated so that it heated the core primarily. The best position was down inside the regular heater as far as the positioning of the RF coil permitted. Under these conditions the core and cladding underwent about 3/4 of the required size reduction before forming an interface. According to theory this should have given ideal preparation of the surfaces. There was some difficulty in obtaining heater balance and cone shape stability, and physical interference from the RF generator limited the experiments to the drawing of 50 cm clad rods. However, many good looking rods were obtained. Transmission measurements, on the rods, however, were disappointing. The attenuation constant was .0040/cm, nearly twice normal. Some of the oversize rods were drawn to longer lengths and retested to check the possibility that surface waviness caused the losses. The results were still disappointing.

Some of the experiments made use of "differential" cladding. In these, the tube was caused to collapse onto the core by feeding it into the furnace at a slower rate than the core. In effect, the cladding tube was drawn at a different drawing ratio such that its inside diameter would have become smaller than the core had the core not been present. No vacuum was necessary or

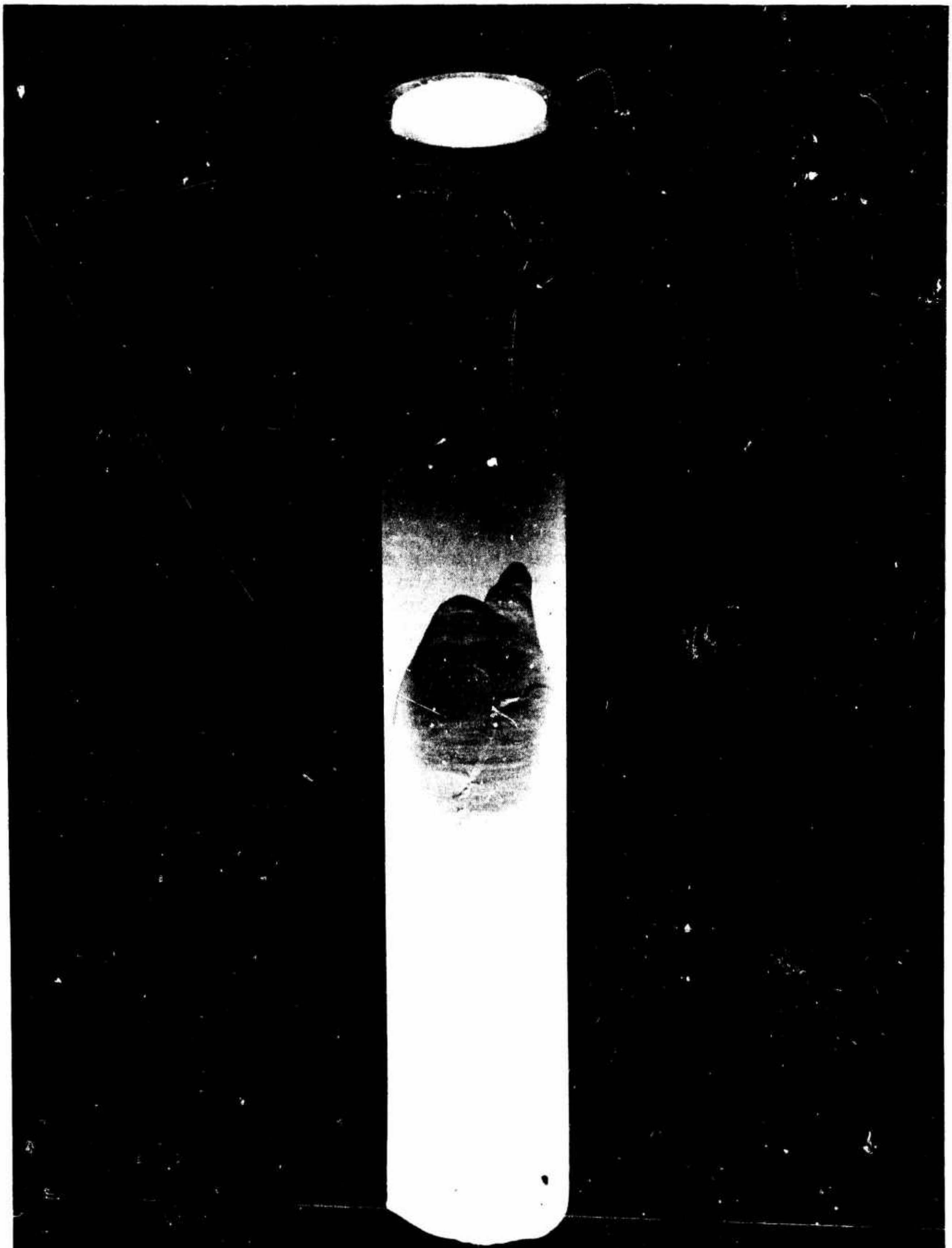

Figure 7 - High Pressure Prefused Assembly

used. This method of closing the cladding onto the core causes the interface to form later or farther down the cone than is the case with the use of a vacuum. This gives more time for baking out and fire polishing the surfaces prior to fusion although the baking is without a vacuum. There is also some drawing of the core and considerable drawing of the cladding prior to fusion. The contact angle between the core and tube is smaller than when a vacuum is used. This may be a drawback. Several variations with differential cladding were tried. Included were F-2 core with R-6 cladding and F-2 core with EN-1 cladding in a standard furnace, F2-R6 with a 150mm preheater above the standard furnace at 400°C and F2-R6 with a 300mm preheater at 400°C. The results were again disappointing, the attenuation constants ranged from .0028 to .0038 per cm versus the .0022 standard figure.

The variations from the normal drawing procedures that were found to be beneficial made use of a heat sink or metal ring to modify the temperature pattern in the upper part of the drawing furnace. It extended into the furnace about 40mm and shielded the glass from direct furnace radiation. The effect was to delay the collapse of the tube onto the core until the glass was farther into the furnace. The objective was to obtain a larger contact angle between tube and core in order to reduce gas trapping. The preheating time and outgassing of the mating surfaces were also improved since the ring became hot enough to act as an intermediate heating zone. Two experiments were run. One used full vacuum (1/10 atmosphere) in the space between the core and tube. The other used a lower vacuum setting (about 1/2 atmosphere). Both experiments gave rods that measured .0020 per cm attenuation. If anything the reduced vacuum is slightly preferable.

One series of experiments evaluated the effect of heat treatment of the clad rods. In these experiments the 3mm F2-R6 clad rods were heated for an extended period after drawing. In most cases the cycle was 19 hours at 600°C. The intent was to diffuse the interface so that the index of refraction gradient would extend over an appreciable thickness. According to the laws of physical optics a gradual change of index can be as effective in causing total reflection as a sharp index change. The path taken by the light, however, will depend on the angle of the ray. Low NA rays, in particular, will penetrate only slightly into the gradient zone. It was considered possible that a significant fraction of the light could be channeled down the fiber without encountering the defects at the original interface, and transmittance would thereby be improved. Initial before and after transmission measurements did in fact indicate some improvement. A more reliable test is the determination of the attenuation constant, although this cannot be run "before and after" because it is a destructive test. Measurements on treated rods indicated

about the same loss constants as standard rods. Measurements on 75 micron fibers drawn from treated rods indicated greater than normal losses (attenuation .0057/cm).

3.0 Tip Fusing

Experiments were undertaken to determine the feasibility of fusing the ends of fiberscopes and, if feasible, to determine what the extent of its usefulness might be. The main advantage of tip fusing is the elimination or reduction of the mosaic pattern due to the cement filled spaces between multifibers in conventional fiberscopes. Further advantages might result from higher temperature tolerance and better cutting and surfacing characteristics. Anticipated problems were the introduction of defects or blemishes due to the fusing step and fragility in the transition zone between the fused and flexible sections. Problems in maintaining fiber alignment and bundle coherency were also expected.

The experimental work in this area showed that it is indeed possible to make optically improved fused tip fiberscopes and that means can be found to overcome the inherent weakness of the transition zones. Although most of the experiments were carried out with small, 3mm, bundles, it was determined that bundles as large as 10mm can be fused successfully. There does not appear to be a sharp limit on the size of bundle that can be fused, but problems do increase with size. In particular, coherency is size dependent due to the greater difficulty of maintaining fiber alignment over the longer fusing zones.

The experiments were carried out primarily with fibers consisting of Schott F-2 core and Kimble R-6 cladding, selected as the most common combination for long fiber applications. The high viscosity of the cladding compared to the core, normally considered undesirable for fusing, did not appear to be a limitation. In fact, experiments with a core harder than the cladding (Kimble EN-1 cladding on Schott UK-50) showed no advantage for that combination. A soft outer coating on the fiber (Corning 7570 solder glass on F2-R6) also failed to be advantageous.

The experimental program started with uncemented and incoherent assemblies of 50μ monofibers placed in a simple 3mm U-shaped mold. Figure 8 is a photograph of an early mold and push block. Heating was by cartridge heaters inserted into the six holes that can be seen in the photograph. The use of the loose fibers made it possible to determine the approximate fusing conditions without complications that might result from the use of a cement. An effort was made to determine the minimum temperature that would be required and the maximum pressure that could be used without longitudinal extrusion. These conditions were found to be 580°C and 140 bars. Figure 9 is an assembly fused

at these conditions. The fact that the fractures follow fiber boundaries rather than cutting across fibers indicates marginal fusion.

The next step was to fuse short ribbon sections of multifibers in the 3mm mold. For this the standard F2-R6 6x6 multifiber design was used, i.e. 60 micron multifibers with 10 micron elements. The ribbons were prepared in the conventional manner except that cellulose nitrate was used as the cement. This cement was intended to burn off completely even in the absence of air. The ribbons were trimmed in width to just fit the die. The fusing cycle was similar to the one used for the uncemented monofibers except for a hold at about 500°C to burn off the cement. This series of experiments showed that the multifibers could be fused in the same manner as the monofibers and in fact bonded together somewhat better at the same temperature. These experiments also showed that the presence of the cement could not be ignored, and that the geometry of the multifiber assembly is even more important in a fused fiberscope than in a cemented one.

Full length ribbons were then fused by the same techniques. These provided an opportunity to evaluate the effect on image quality of defects due to the cement, irregularities and spaces in the ribbons and fiber misalignment. The relative freedom from major blemish problems at this stage was quite encouraging.

The first attempts to overcome the effect of spaces between fibers in the ribbons were made with a type of "two-way" squeeze. Ribbon sections were trimmed to a width about 0.4mm wider than the mold. The side plates were loosened enough to permit assembly. When the fibers were at fusing temperature the initial pressing motion was from the side and compacted the fibers by this 0.4mm. The top plunger was then moved to complete the pressing in a vertical direction. Examination of the bundle showed no advantage for this procedure, and it was decided that problems due to spaces would have to be solved by improving the precision of the ribbons.

In the next experiments an effort was made to overcome the fiber breakage problem in the transition zones between the fused and unfused sections. This was done by potting the fibers in cement in these zones. The procedure is shown in Figures 10 and 11. As indicated in Figure 10, the ribbons were prepared with three bands of cement, a central band of cellulose nitrate and two outer bands of "B" stage epoxy. The epoxy bands help to maintain fiber alignment outside the fusion zone and permit the use of a narrow band of cellulose nitrate. Their main functions, however, are to support the fibers after fusing and to act as a dam to control the flow of the low viscosity epoxy wicked into the fibers in the transition zone. The procedure used was to cure the "B" stage epoxy under low pressure in a long mold. The bundle was

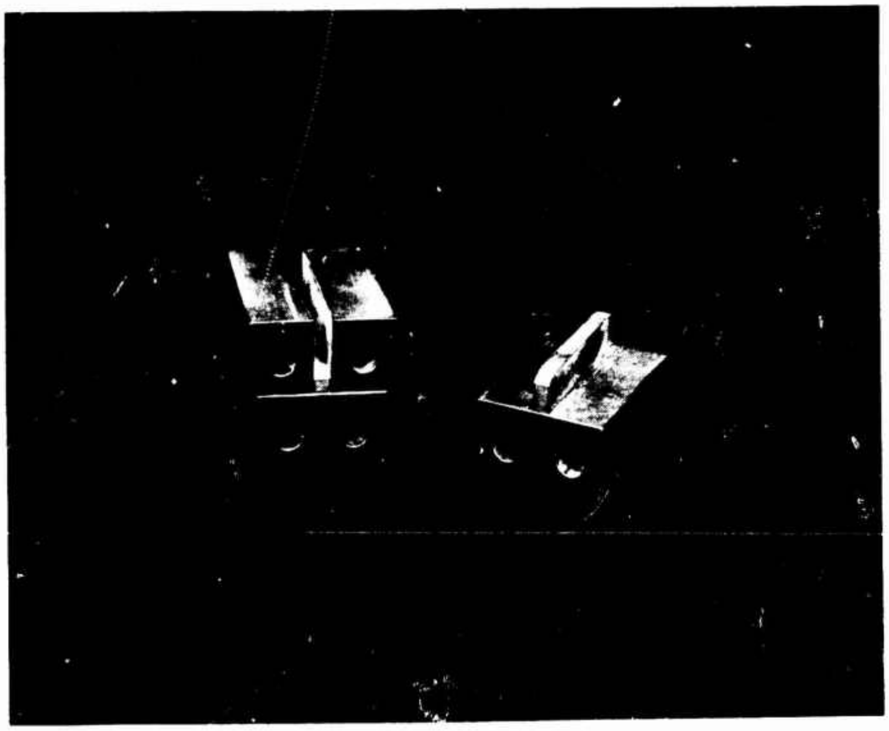

Figure 8 - Simple End Tip Fusion Die

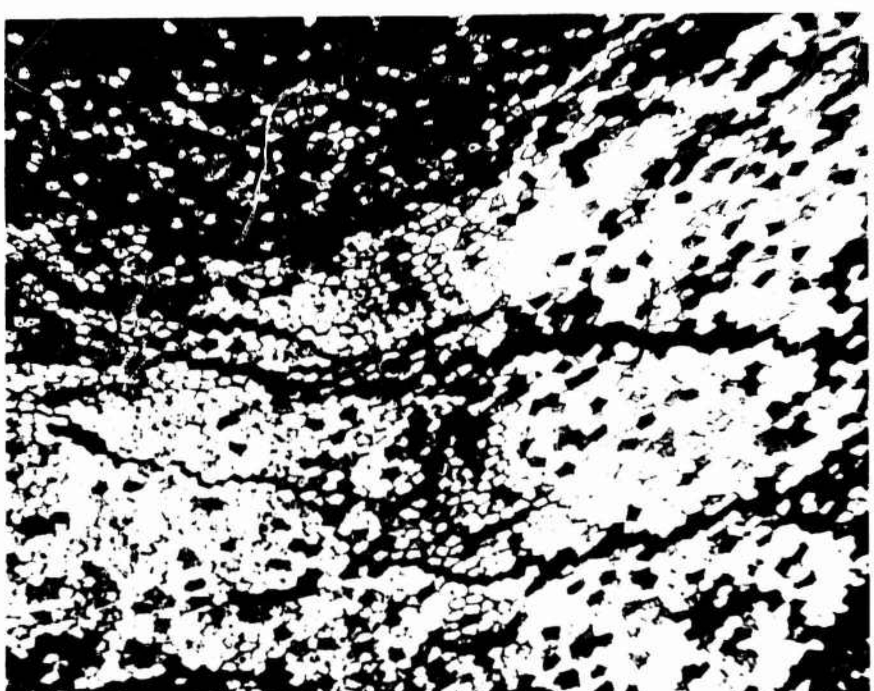

Figure 9 - Cross Section of Non-Orientated Fiber Fusion Using Simple Die (See Figure 8)

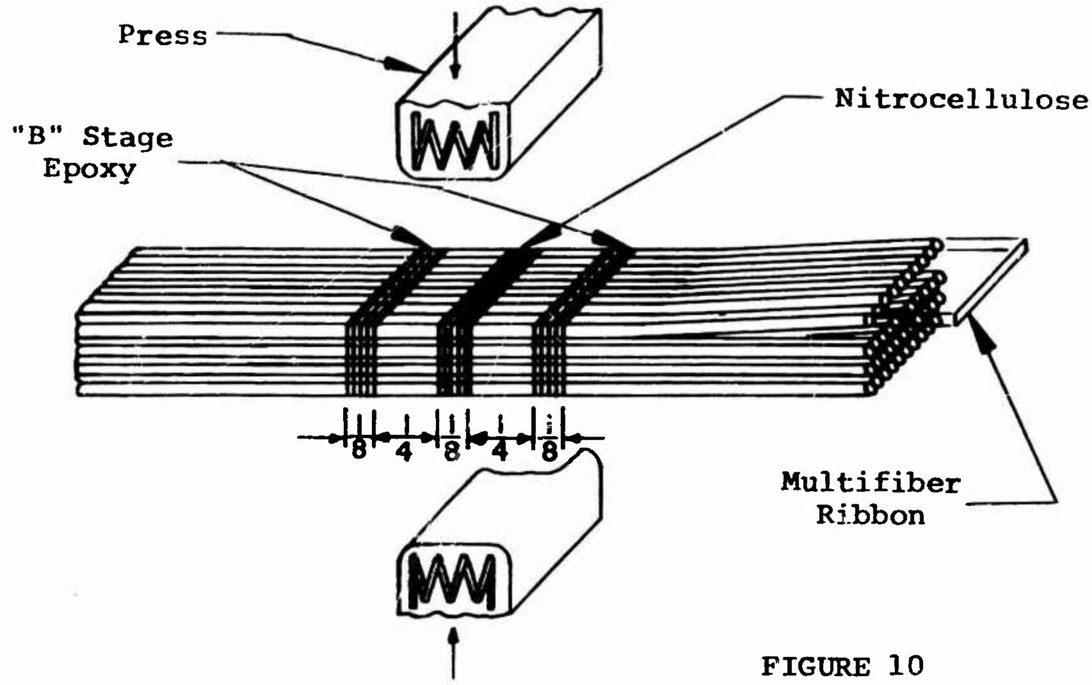

FIGURE 10
PREFUSED RIBBON STACK

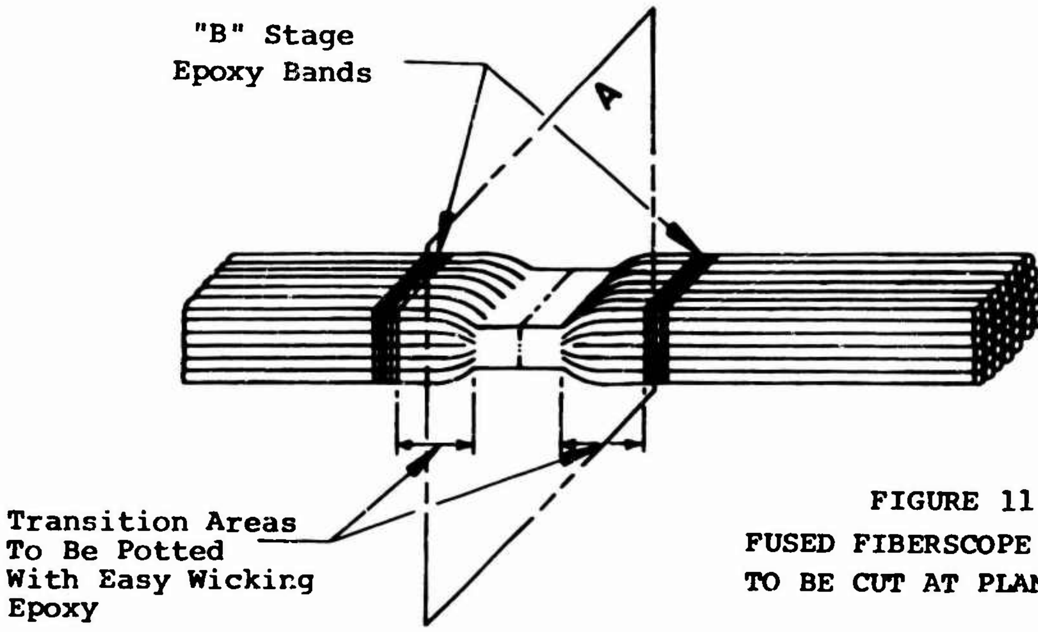

FIGURE 11
FUSED FIBERSCOPE SECTION
TO BE CUT AT PLANE "A"

then transferred to a shorter mold and fused in the same manner as previous bundles. After fusing, the low viscosity epoxy was wicked into the fibers between the fusion and the "B" stage bands, excessive wicking along the fibers being prevented by the bands. This technique in combination with a refinement in fusing mold design appeared to solve the fiber breakage problem in the transition zone. The refinement was to flare the edges of the mold on all four sides to minimize stresses on the outer fibers. Previously only the top and bottom had been flared. With these changes the breakage problem appeared no worse than for conventional cemented fiberscopes. Figure 12 shows a fiberscope in the cement curing mold. Figure 13 shows the bundle in the fusing mold. Figure 14 shows the spacing of the fibers in the "B" stage epoxied section. Figure 15 is a micrograph of a thin slice from the fused section.

In addition to reinforcing the transition zone, other experiments were performed with the experimental bundles at this stage. One series was aimed at determining the optimum pressing conditions for multifibers. These determined that a high pressure is desirable during the compaction period (the 140 bar value selected for monofibers is satisfactory), but it is desirable to stop pressing just prior to reaching the full density. The high pressure during pressing is to cause the compaction to take place in minimum time for minimum bubble growth. The pressure, of course, must not be high enough to fracture or otherwise damage the fibers. A stop was provided by the mold, and the density was controlled by the degree of fill. Best results were obtained with about a 96% fill. The firm stop provided by the mold also gave a good indication of when the pressing cycle was complete. Thus, when the plunger travel ceased, the heat was turned off quickly, minimizing bubble growth.

At this stage in the project experiments were also performed in an effort to eliminate blemishes that seemed to be due to the cement. In these the cellulose nitrate band was eliminated completely. At the time, however, this did not appear to be desirable. Bundle geometry and coherency suffered, and not all of the cement related problems seemed to disappear. It was concluded that although some of these problems were due to imperfect combustion of the cellulose nitrate, others were due to the epoxy. The epoxy problems were due to some of the ribbons being positioned at assembly with cement too close to the fusing zone and to flow of epoxy along the fibers as it softened just prior to polymerization during the curing cycle.

Steps were first taken to improve the precision of cement application and ribbon stacking. This was done with improved fixturing and by coloring the cement to improve its visibility. Then the process was altered to eliminate the possibility of flow of the epoxy into the fusing zone. To do this the fusing mold was modified to provide a controlled temper-

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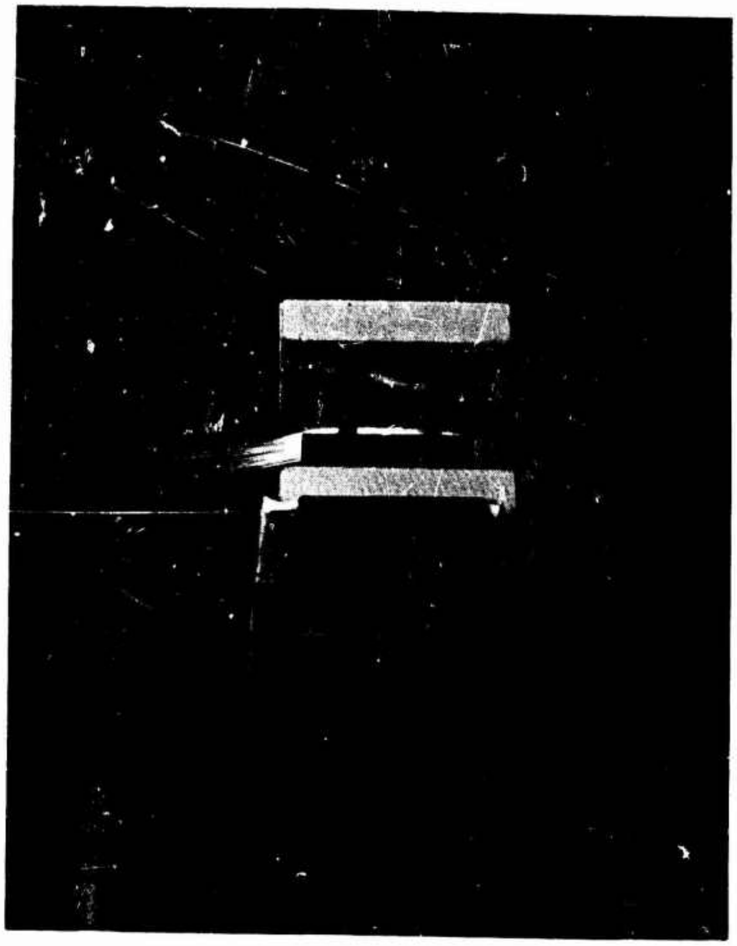

Figure 12 - Stacked Bundle in Position for
Curing of "B" Stage Epoxy

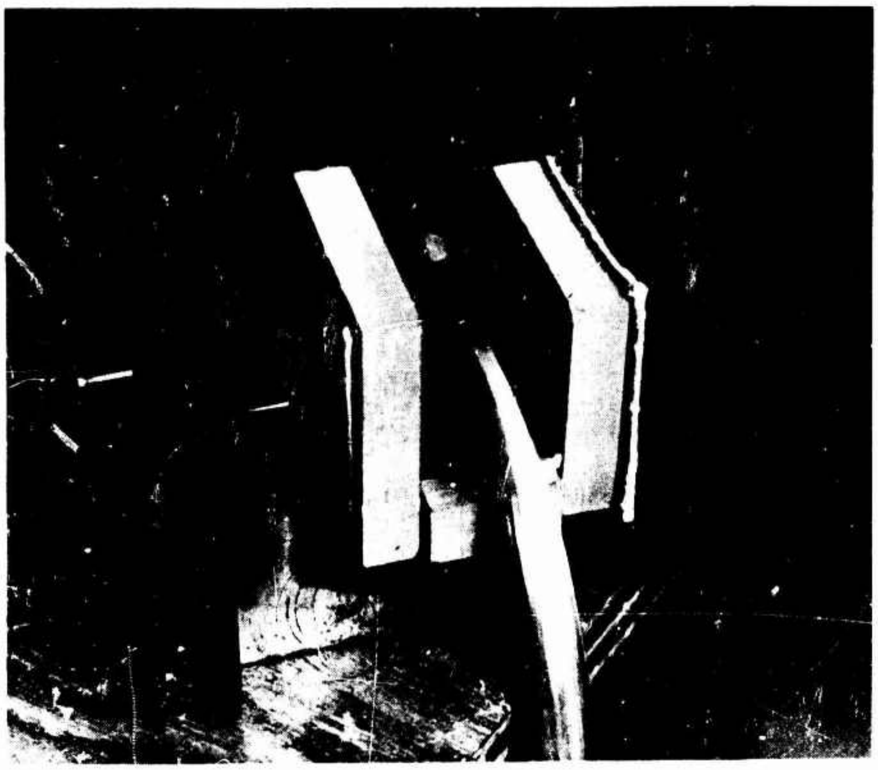

Figure 13 - New Short Zone Die And Bundle Ready For Fusion

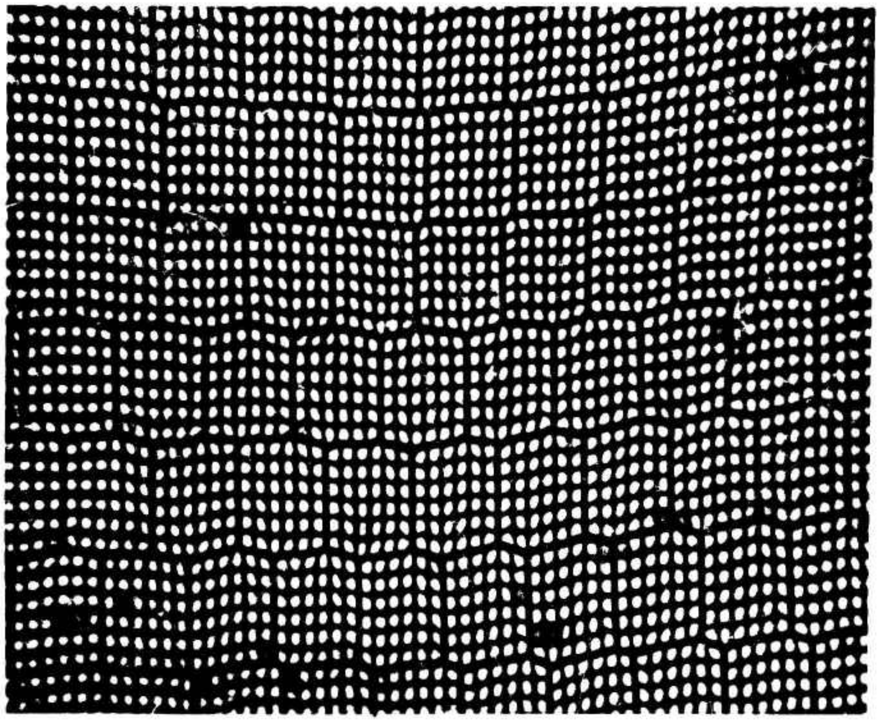

Figure 14 - Photomicrograph of Cross Section of Same Bundle Through "B" Staged Area

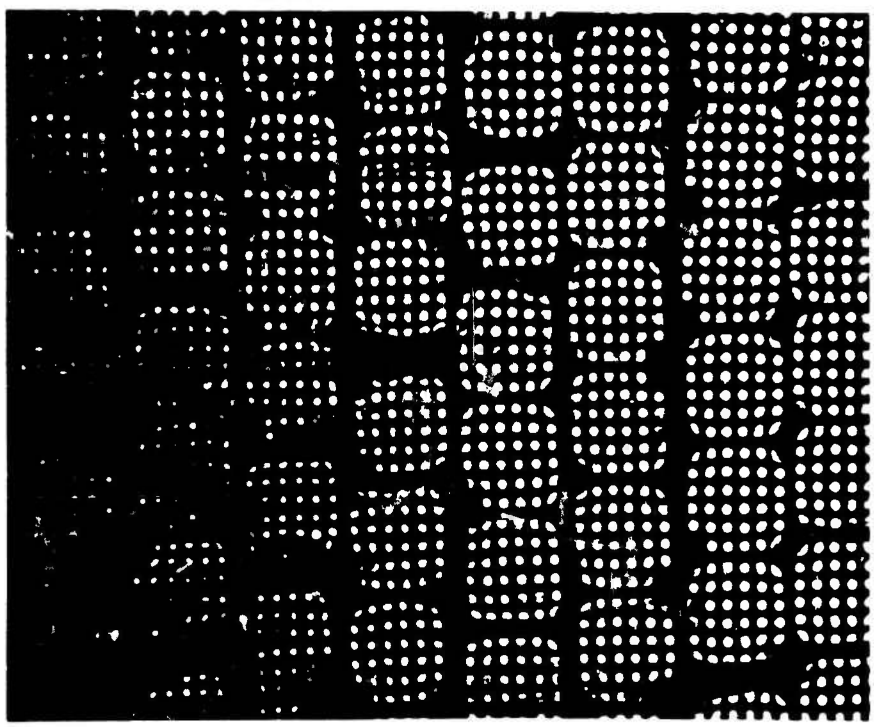

Figure 15 - Photomicrograph of Cross Section of a Fused Bundle

ature gradient along the fibers so that the epoxy could be cured during the fusing cycle. Figure 16 shows the modified mold, partially assembled, with the fibers in position for fusing. This technique of simultaneous fusing and curing was very successful in controlling the flow of the epoxy, and it was used in all subsequent fusions. The temperature gradient along the fibers completely prevented the wicking of epoxy into the fusing zone.

The next step in the development of tip fusion was in improving the precision of the assemblies to be fused. Spaces within the ribbons were reduced by more careful hand packing. The width of the ribbons was controlled more precisely by going to exact count winding instead of slitting the ribbons to width. The elimination of voids in the assembly gave a significant improvement in bundle appearance and coherency.

With these improvements in ribbon precision an attempt was again made to eliminate the cellulose nitrate cement. This time it was successful. Satisfactory fiber positioning and alignment was maintained across the 14mm gap between the two epoxy bands.

A series of 3mm fiberscopes was made to test the repeatability of what at this point appeared to be a satisfactory tip fusing process. Unfortunately, they were not successful. Figure 17 shows the blemish content of a typical sample. To determine the location of the blemishes some fiberscopes were cut into four pieces as indicated in Figure 18. Figure 19 is a view through the central 2mm slice from the fusing zone and is quite free of blemishes. Figures 20 and 21 are views through one of the transition zone pieces as seen from the fused end and the cemented end respectively. This section is also quite free of blemishes as was the corresponding piece from the other end of the fiberscope. Figure 22 is a view through the remaining 98cm of the fiberscope between the two cemented zones and is seen to contain most of the blemishes. Evidently this group of samples was made inferior multifiber stock. This was not discovered until the series was completed; however, examination of the fused ends of all of the samples indicated that the fusing process is quite reliable for the 3mm size assemblies.

On this basis it was decided to explore the possibility of fusing larger bundles. The size selected was 10mmx10mm. Fiber was prepared for four fusions at this size. Three were in the form of short ribbon sections to be used in evaluating mold and ribbon designs. The fourth was a full length fiberscope.

The first large fusion was a simple scale-up from the smaller fusions; i.e. the length of the fusing zone was increased in proportion to the increase in size. This gave a heater length of 38mm and a total distance between epoxy bands of 45mm. Because

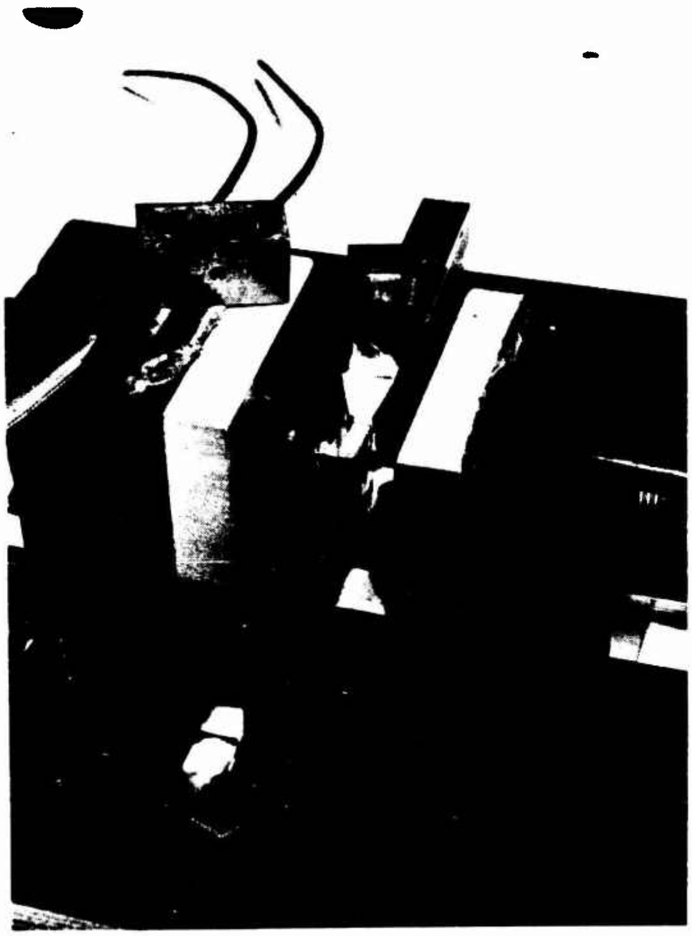

Figure 16 - New Tip-Fusing Die Containing a Short Fiber Bundle Section. The Bundle Outboard of the Pressing Pieces is Encased in Aluminum Heat Radiators That Also Compress the "B" Stage Sections

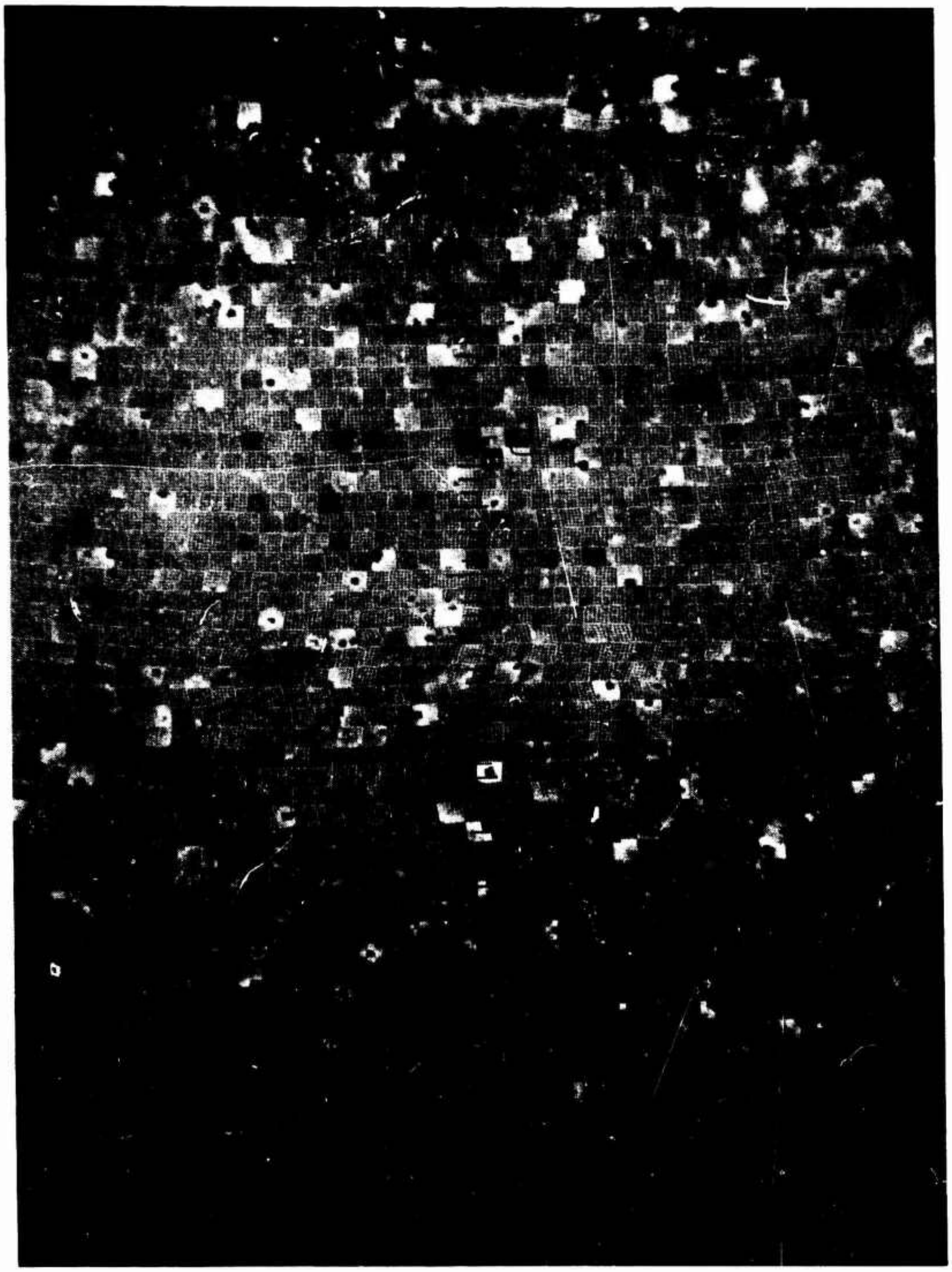

Figure 17 - View Through Full Fiberscope as Fused to Test Repeatability - F2-R6, 3 x 3mm Cross Section

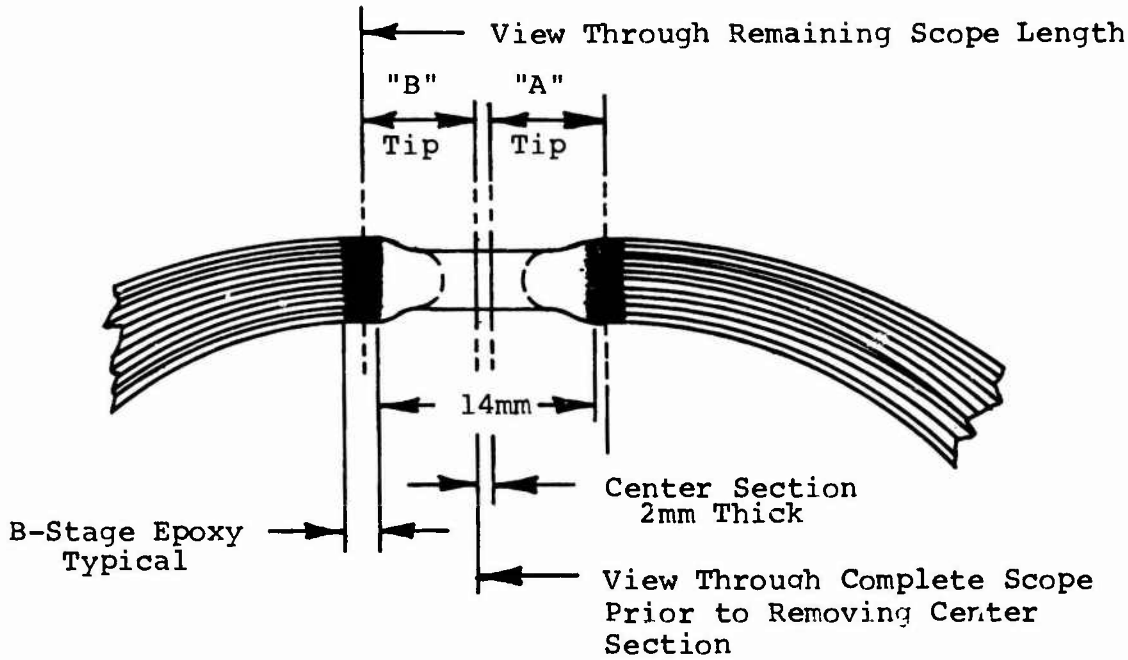

Figure 18 - Dissecting Technique Showing Location of Cuts in Fused and Cemented Areas of Fiberscope

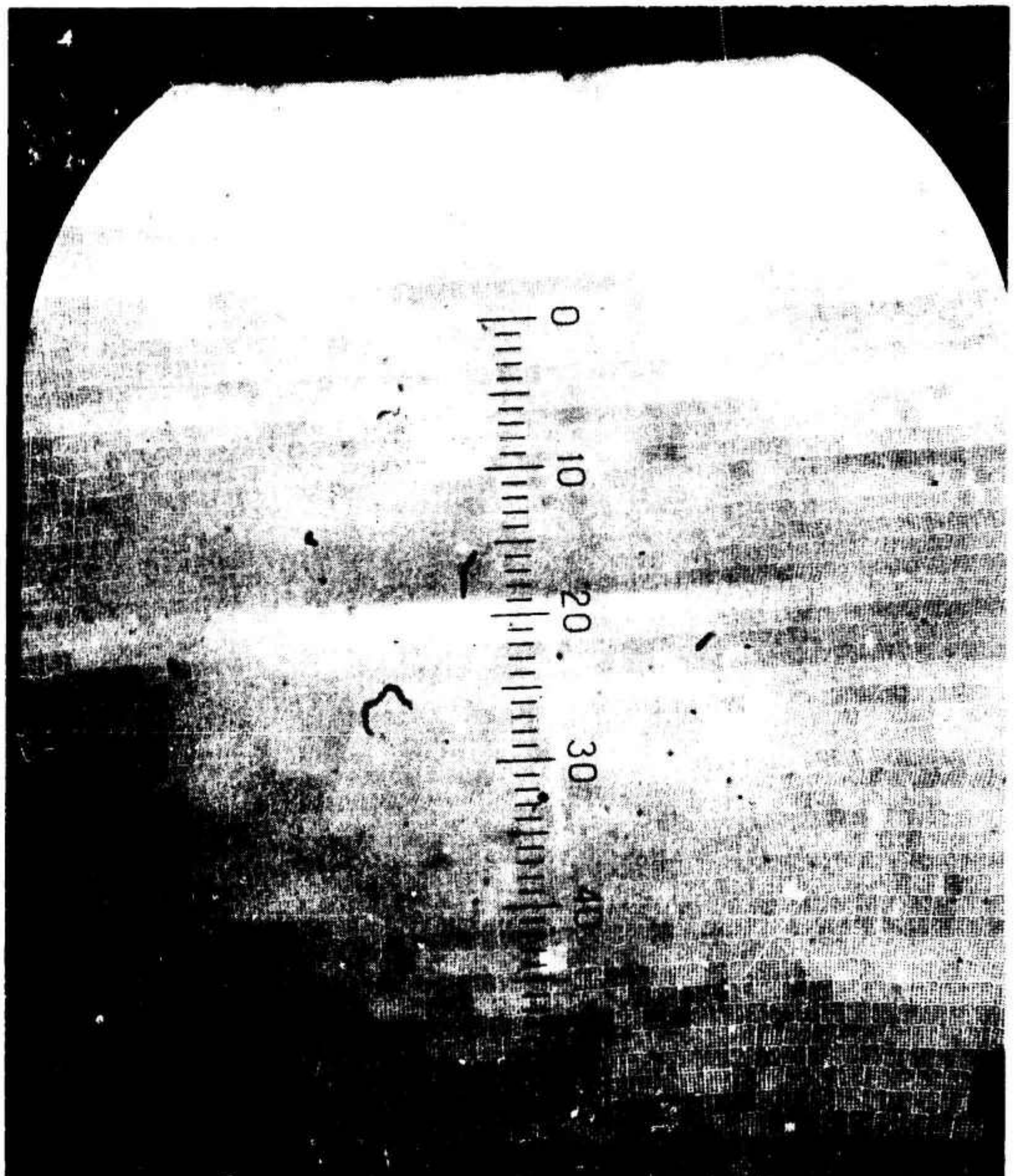

Figure 19 - Central 2mm Slice Through Fused Section of Fiberscope Shown in Figure 17

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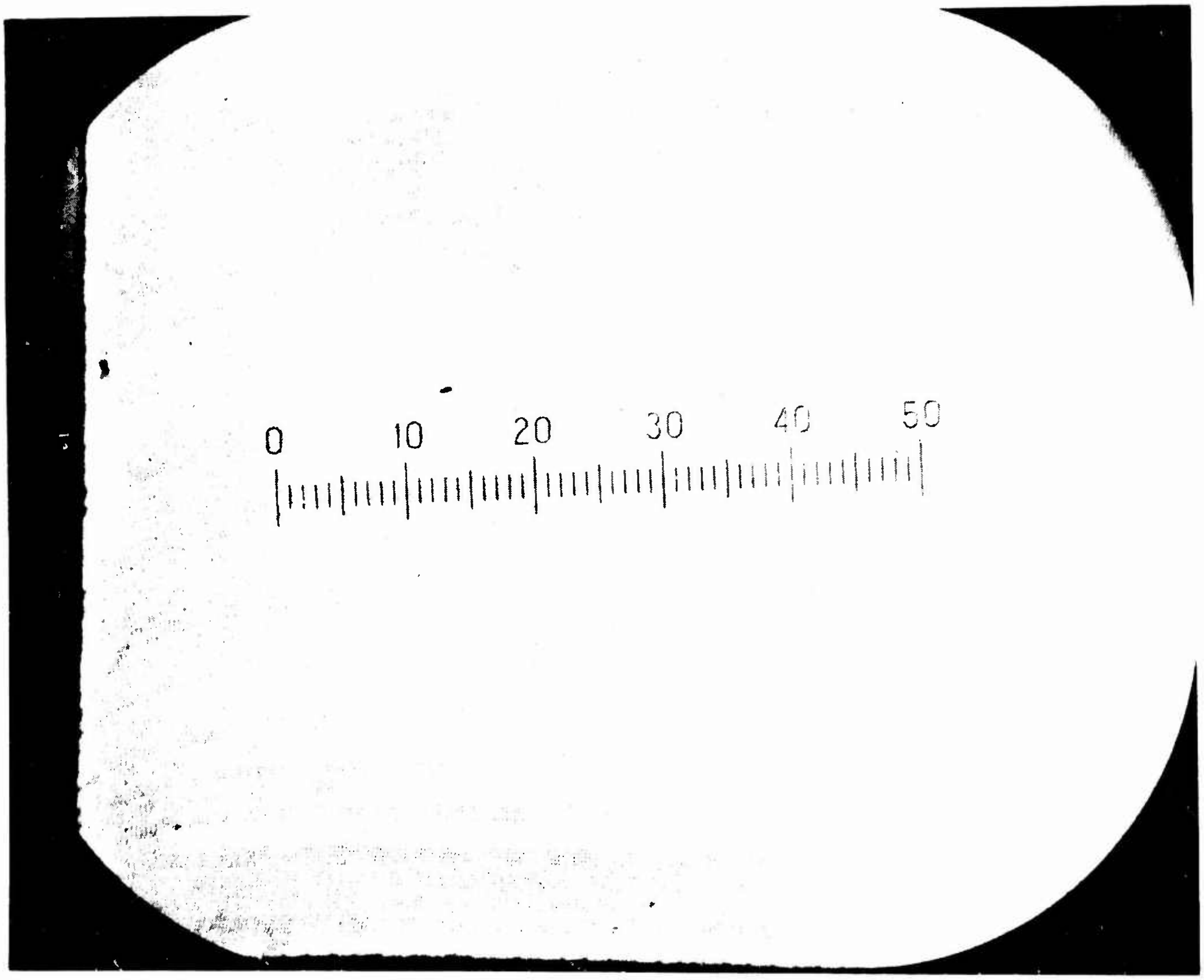

Figure 20 - View Through Transition Section - Fused End Forward

Figure 21- View Through Same Transition Section - Epoxy
Reinforced End Forward

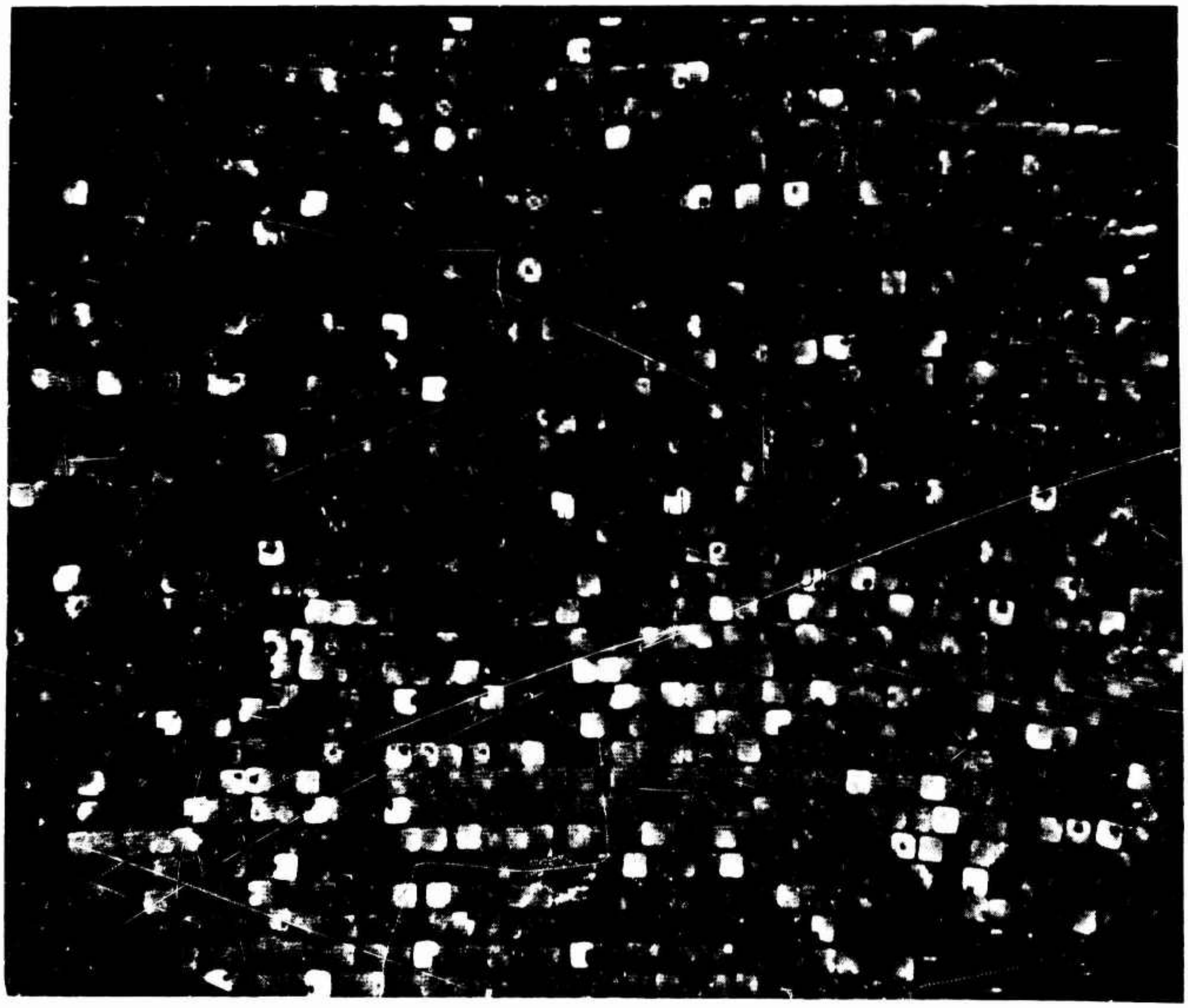

Figure 22 - View Through 35" Unfused Portion of Same Bundle

of the increased fiber length between epoxy bands it was anticipated that coherency would be a problem unless some support was provided. Two bands of cellulose nitrate were therefore added between the epoxy bands leaving about the same length of unsupported fiber as in the smaller bundles. The dimensions are shown in Figure 23. To minimize problems from the added cement a very dilute mixture was used. Fusing and epoxy curing were carried out simultaneously as in the smaller bundles. Pneumatic cylinders were added to the press to apply more force to the cemented sections during curing than was feasible with the weights that were used previously. The fusing equipment used for the 10mm size is shown in Figure 24.

The results of the first fusion were encouraging except for some multifiber misalignment. Figure 25 shows a central slice from the fused section. The effects of misplaced fibers can be seen. Figure 26 is a magnified view of the same slice showing the displaced fibers in greater detail. Figure 27 is a view of a selected section of the central slice and shows the quality that is desired.

To improve fiber alignment the use of cellulose nitrate was increased in the second 10mm fusion. A more concentrated solution was used, and the bands were made wider, reducing the unsupported section to 6mm (indicated by the dotted lines in Figure 22). This appeared to solve the fiber alignment problem but reintroduced the problem of blemishes due to the presence of the cement. Figure 28 is an overall view through a central 8mm slice from the fusion and shows the substantial incidence of blemishes. Figure 29 is an enlarged view of a thinner (2mm) slice and shows two "line" blemishes that are believed to be due to cement. Figure 30 shows a blemish at an even higher magnification. The presence of foreign material is indicated by the space between multifiber layers.

In the next experiment the possibility of using a much shorter fused section was explored. The heater length was reduced to 16mm, and the cellulose nitrate was eliminated entirely. This proved to be too short for the 10mm cross section, the center being under-fused.

Based on the results of the third fusion, compared with the first two, it was decided to select an intermediate fusion length for the full fiberscope experiment and to fuse without cellulose nitrate. A heater length of 21mm was selected. The resulting fused fiberscope was disappointing, however. A view through the full one meter length is shown in Figure 31. The high incidence of breakage is due to an accident in handling and is not related to the fusing step. The breakage masks some of the other defects. To better understand these defects the fiberscope was cut through the cemented zones. Figure 32 is a view through one transition zone. This shows substantial areas of

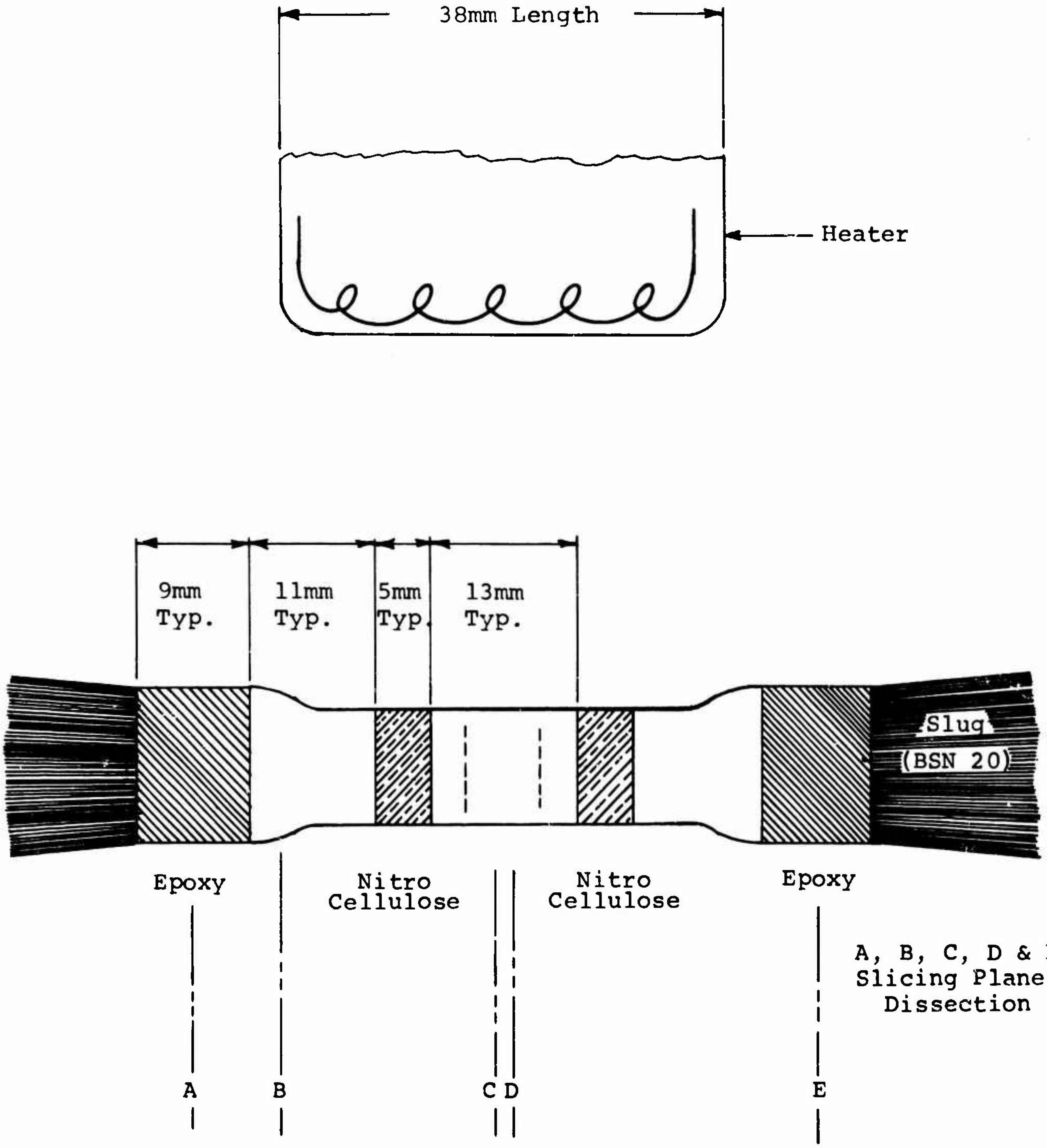

Figure 23

Scale Drawing For 10mm Fusing Proportions

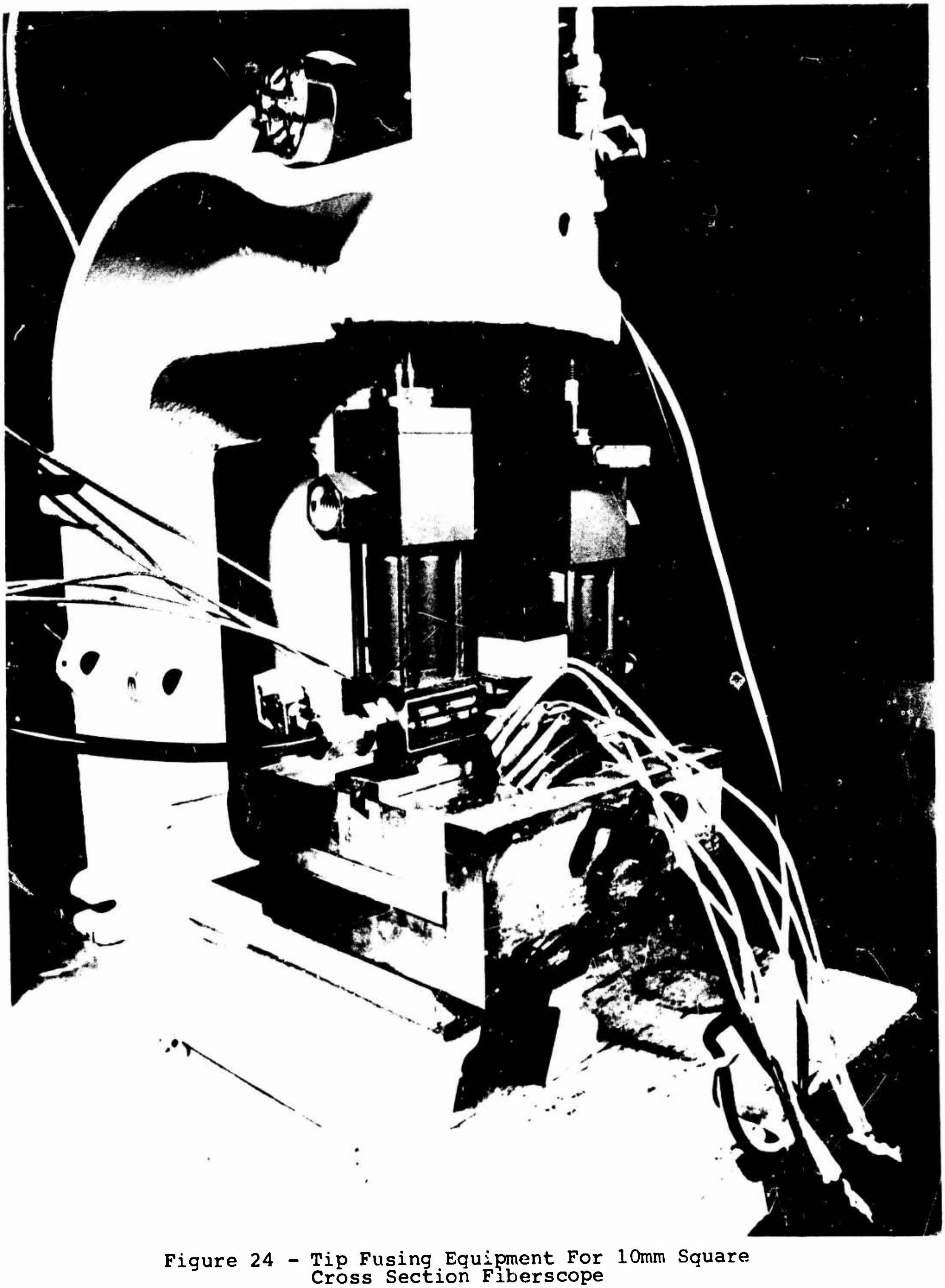

Figure 24 - Tip Fusing Equipment For 10mm Square Cross Section Fiberscope

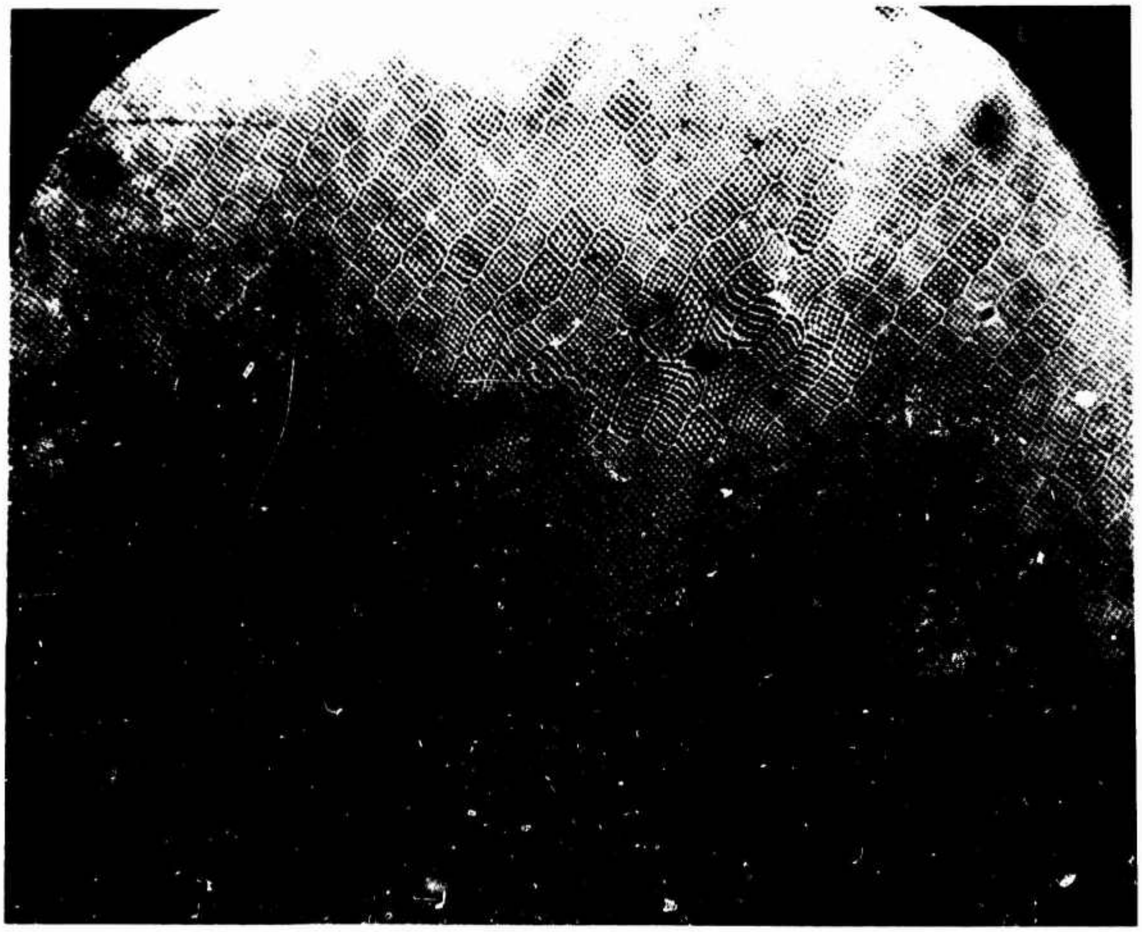

Figure 25 - 1.3mm Center Slice

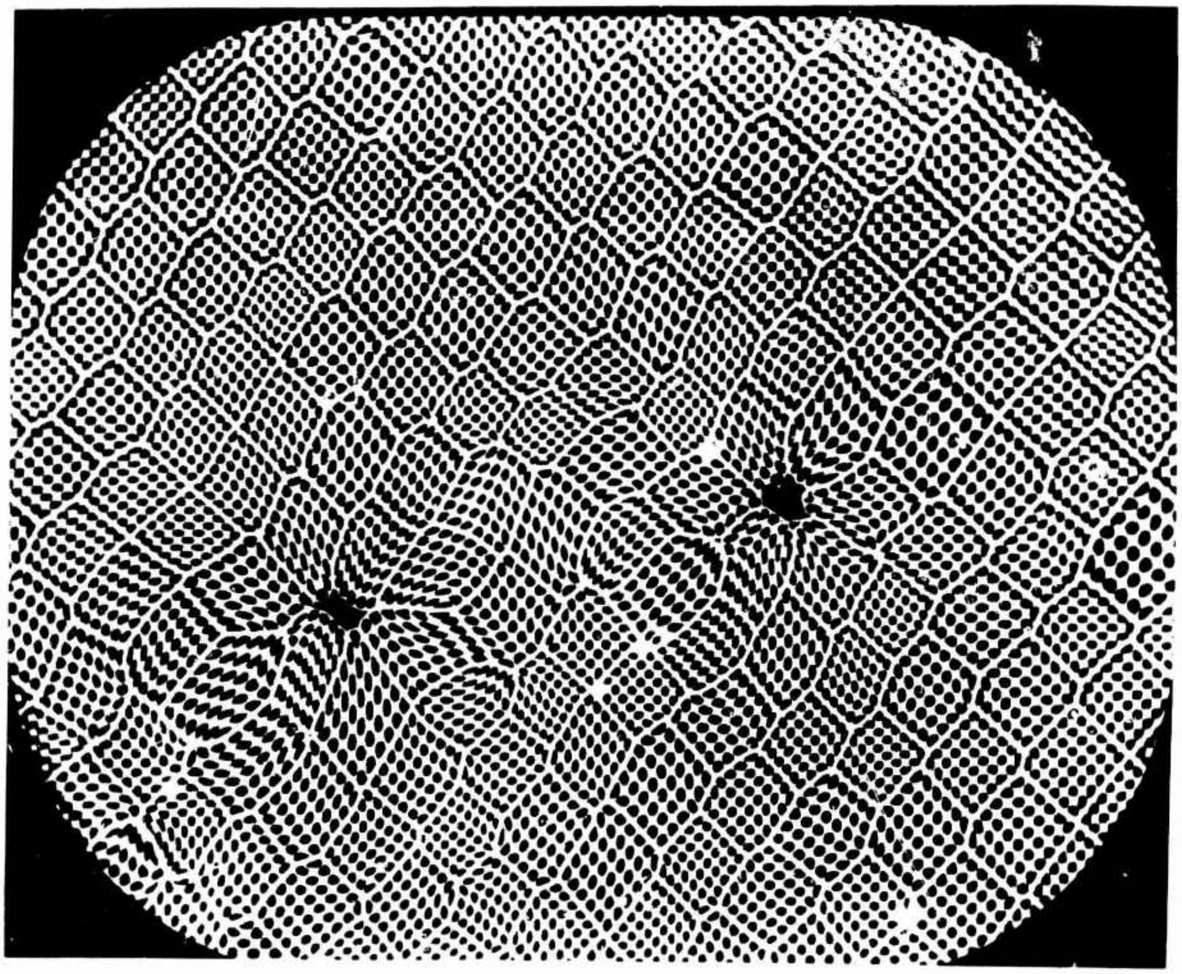

Figure 26 - 1.3mm Center Slice

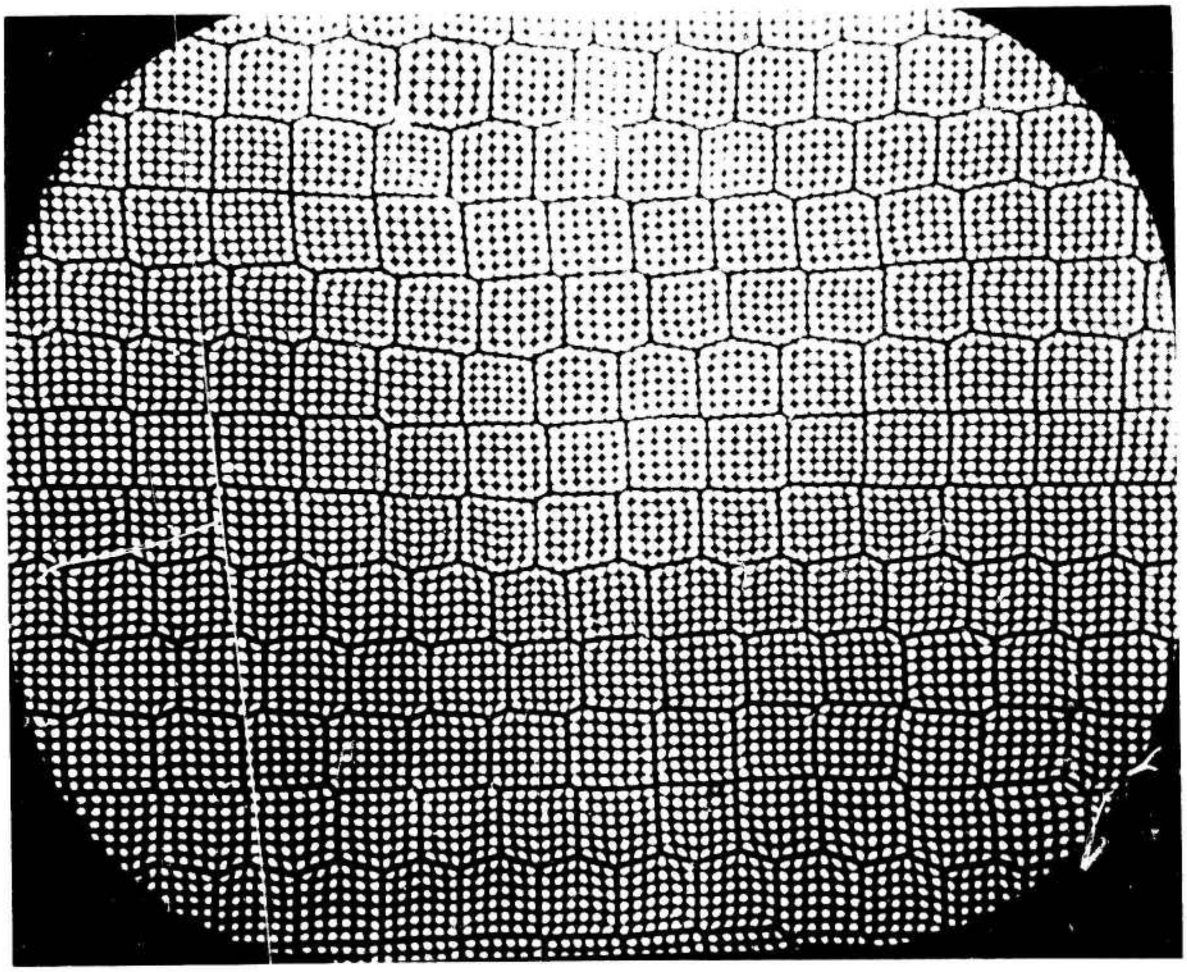

Figure 27 - 1.3mm Center Slice (Selected)

NOT REPRODUCIBLE

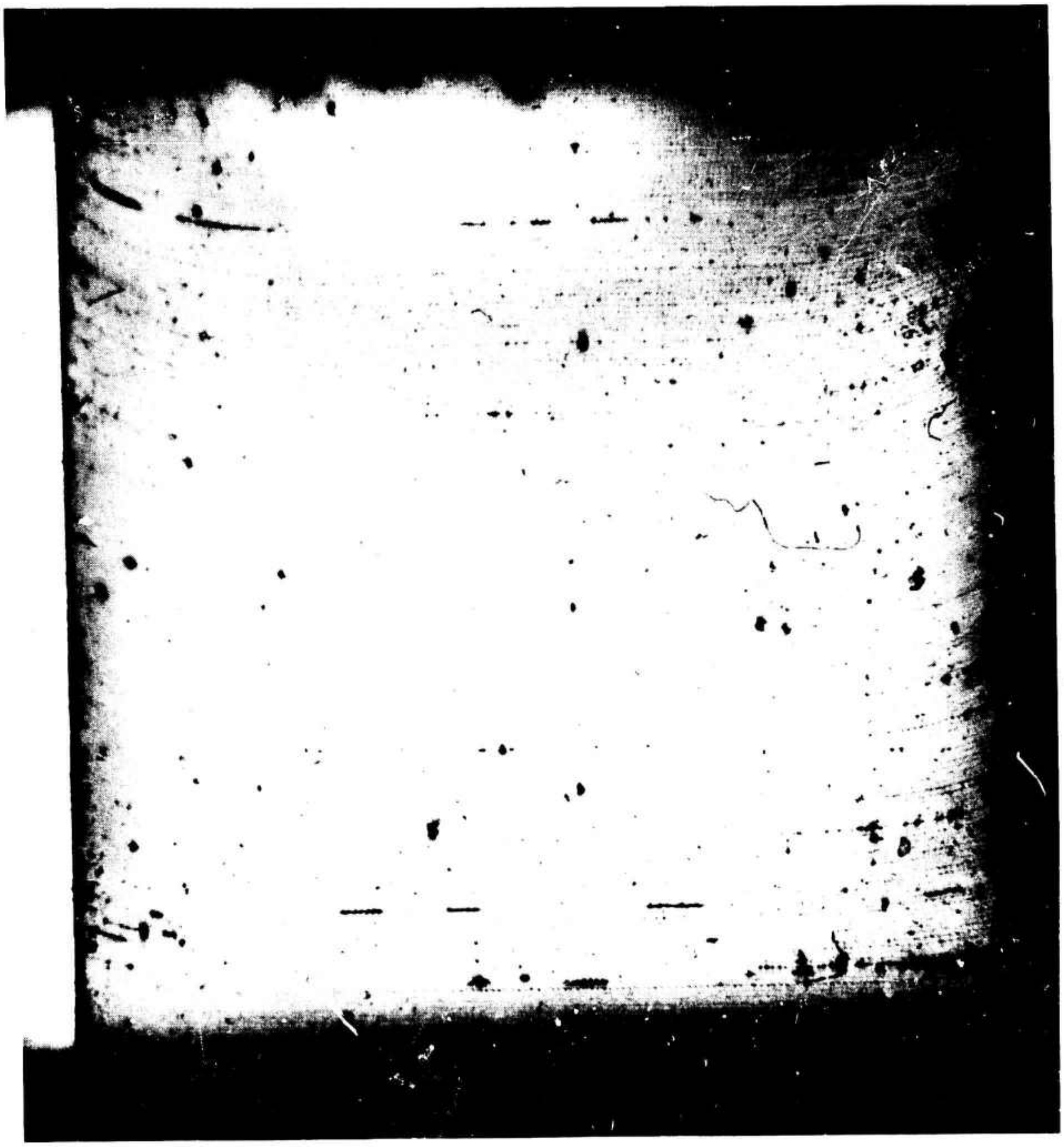

Figure 28 - 8mm Center Slice

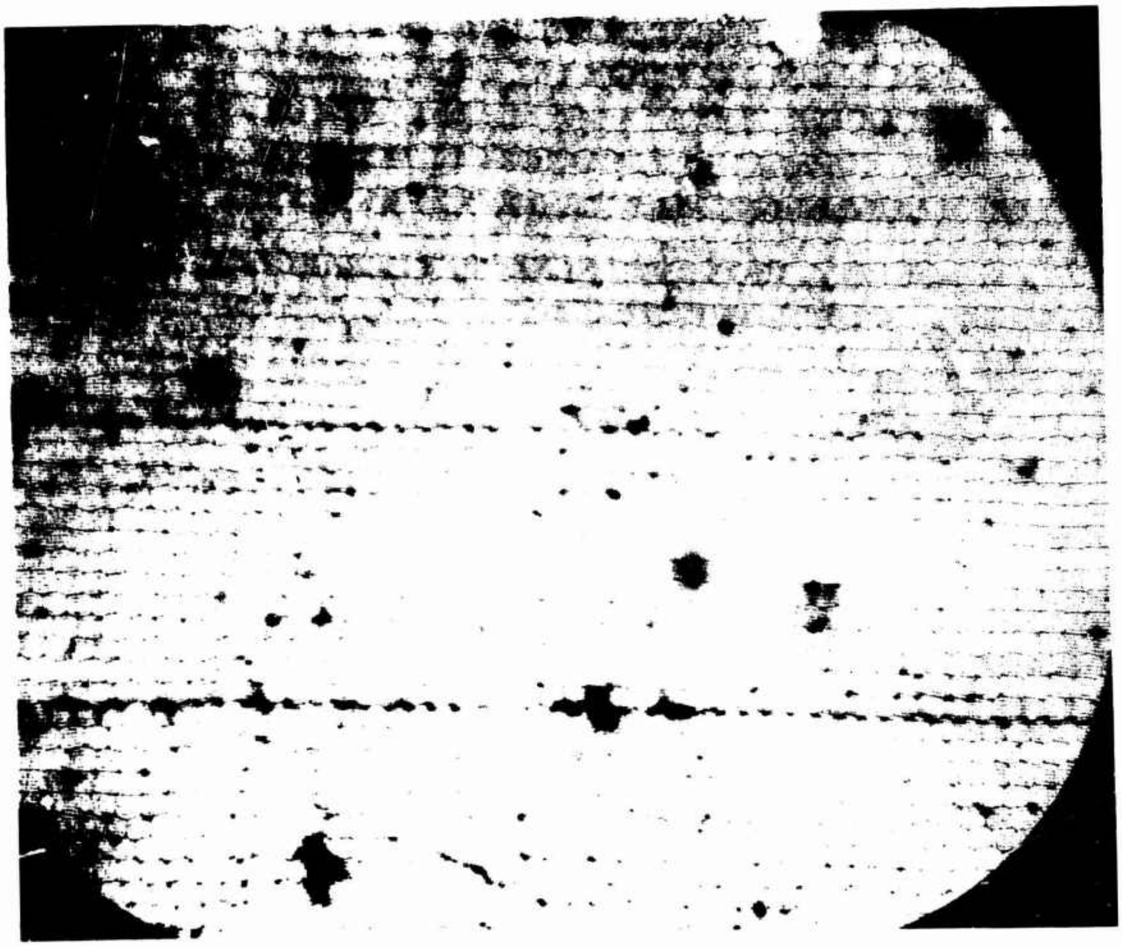

Figure 29 - 25mm Center Slice (Typical of Nitrocellulose)

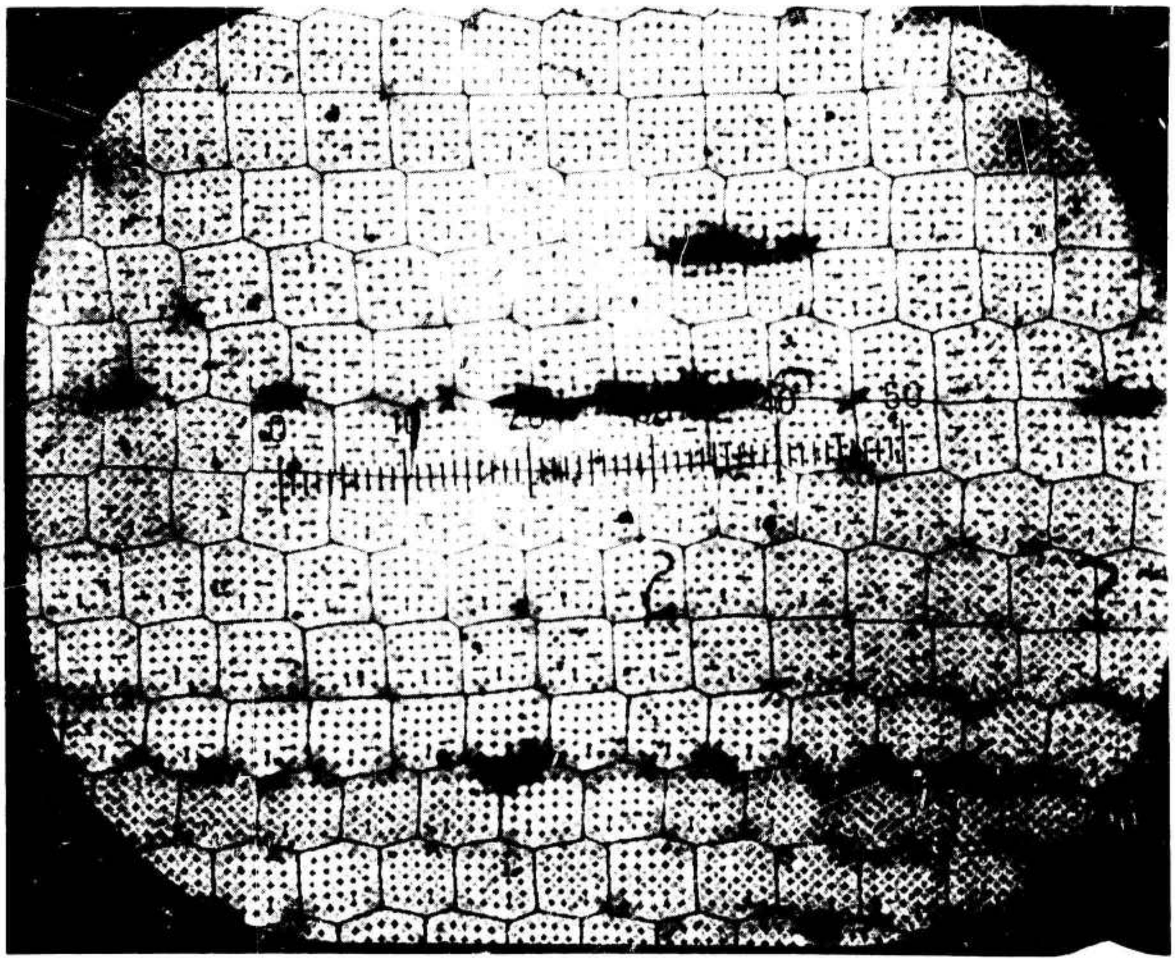

Figure 30 - Example of Nitrocellulose Entrapment

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Figure 31 - Cross Section View Through
10mm x 10mm x 1 Meter
Experimentally Fused Fiberscope

Figure 32 - Transition Zone of 10mm x 10mm Fiberscope Viewed From Fused End

good fusion in the upper portion but some very bad spaces and areas of dislocated multifibers in the lower portion. In addition, the lower portion is incompletely fused. The incomplete fusion is attributed to unusual multifiber size variation which occurred during drawing of some of the ribbons due to a winding machine malfunction. This was not discovered in time to prevent their inclusion in the assembly or to permit proper allowance to be made for the variation in predicting overall packing density. However, the incompleteness of the fusion actually helps in analyzing the defects. Further compacting would have made them less conspicuous but would not have eliminated their adverse affec on coherency.

A possible explanation for the defects would be that the 21mm unsupported length for the multifibers is too long whereas 14mm and 16mm lengths used previously were satisfactory. However, the defects seem too severe to be completely explained in this way. One possibility is that the fiber size variation caused an unusually bad condition in the way forces were transmitted through the assembly during pressing. The cemented ends of the transition zone samples have been examined for possible substantiation of this possibility. The size variation is not particularly obvious at low magnification as in Figure 33. However, under higher magnification local areas can be found where oversize fibers in certain rows occur immediately above oversize fibers in other rows. One instance is shown in Figure 34. Such occurrences could provide the basis for the large dislocations found in this bundle.

The results on the four experiments at the 10mm size indicate that a large number of experiments would be required to define a reliable fusing procedure for that size. The likelihood of eventual success must be considered good, however, in view of the good quality of much of the last bundle and the probability that the major defects are not related to bundle size.

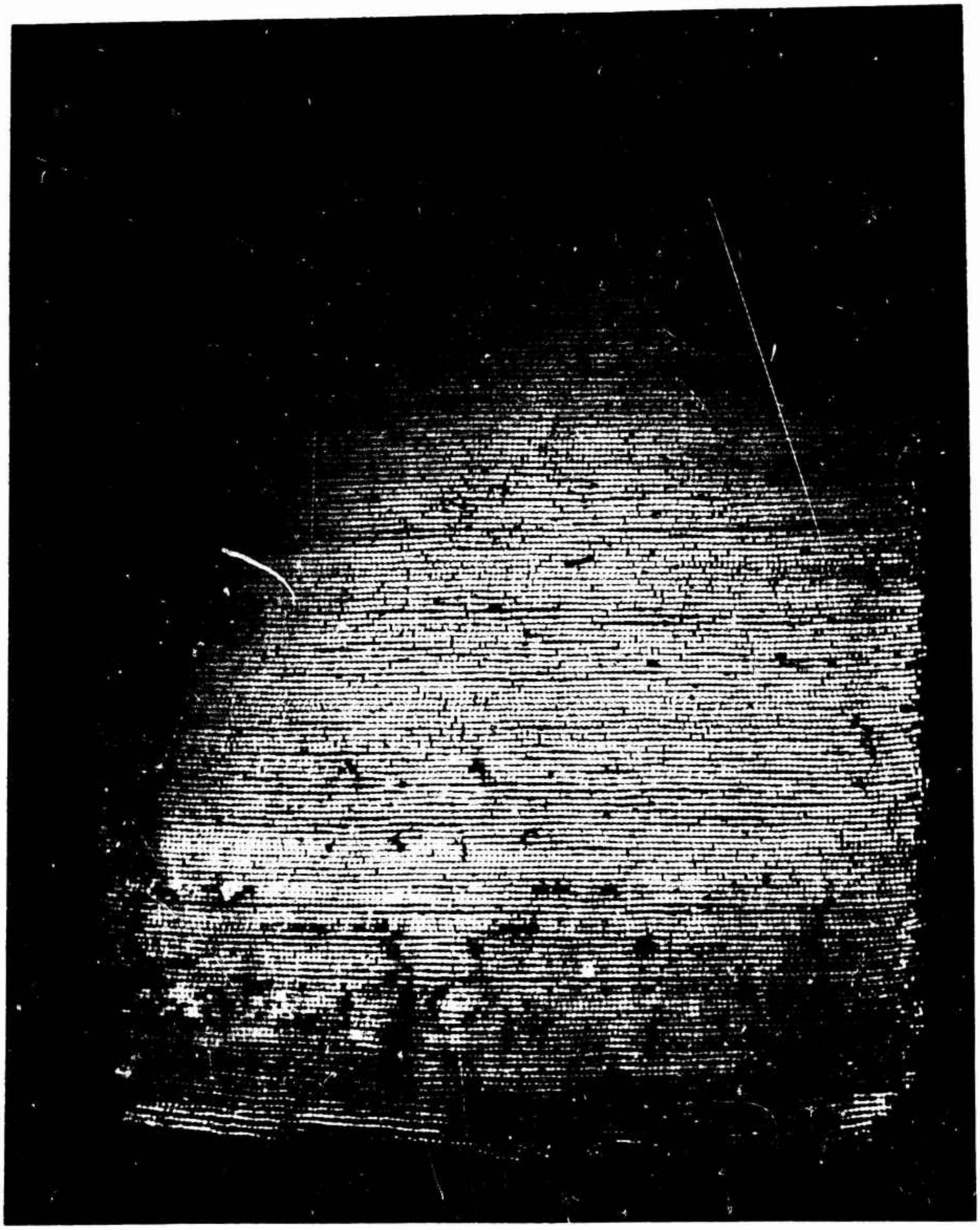

Figure 33 - Transition Zone Of 10mm x 10mm Fiberscope Viewed From Cemented End

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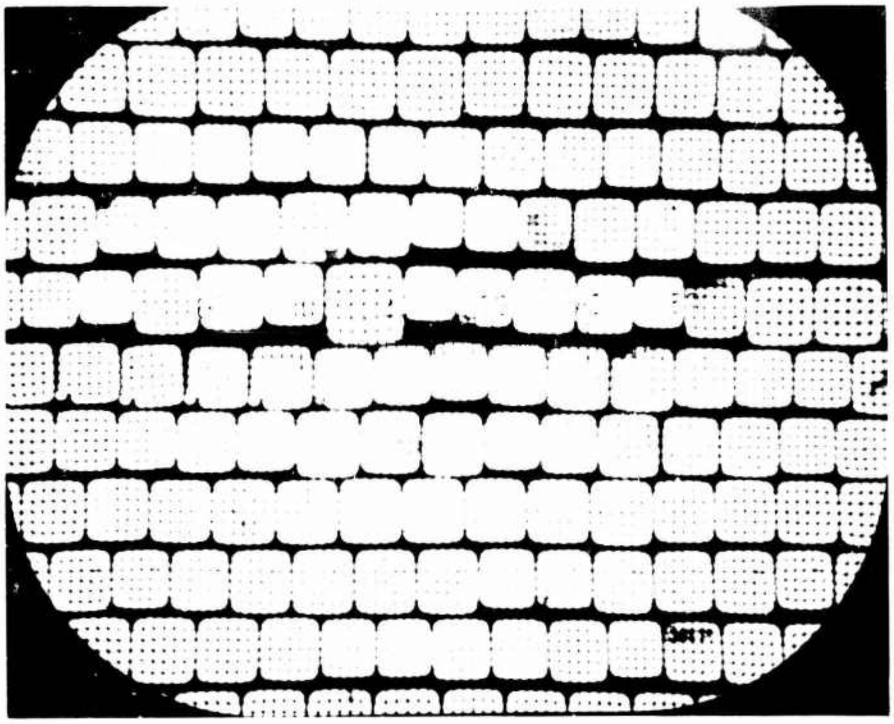

Figure 34 - Area in Cemented Section of
10mm x 10mm Fiberscope Showing
Fiber Size Variation

4.0 Experimental Drawing

4.1 Introduction

This section covers those drawing experiments not described in the other sections. These include some experiments to improve the processing of conventional materials but is mainly concerned with the special problems of new materials. Based on the selection techniques given in the following section a total of 10 core glass and six cladding glass candidates were evaluated by incorporation into clad rod. Special attention was given to determining the feasibility of using a fluorocrown cladding.

4.2 Core Glasses

The core glasses selected were Schott UK-50, LF-5, BaF-4, F-6, F-7, BaK-1, SF-1, SF-4, Ohara 620-362 and Corning 8378. To maintain standard evaluating conditions, Kimble EN-1 or its equivalent, Corning 7052, was chosen as the cladding for each candidate unless problems developed which required an alternate choice.

Six of these glasses were successfully incorporated into 3mm rod on the first attempt using a cladding of Kimble EN-1 or Corning 7052. These included Schott UK-50, LF-5, BaF-4, F-6, Ohara 620-362 and Corning 8378. In addition, a rod was drawn using a standard Schott F-2 core and Corning 7052 cladding.

Schott SF-4 and Kimble EN-1 were drawn into 3mm rod; however, the rod contained an excessive amount of bubbles and the transmission was very poor. The experiment was repeated with a Kimble R-6 cladding and the transmission was improved; however, bubble content remained excessive. A final experiment using a round core bar produced rods with a somewhat higher transmission, but it appears that bubble formation in this glass makes it unsuitable.

An attempt to draw Schott SF-1 with a Kimble EN-1 cladding proved unsuccessful due to the low softening point of the SF-1. The result was misshaping of the core and subsequent peeling of the cladding.

Rods drawn with a Schott F-7 core and Kimble EN-1 clad broke up due to high internal stress introduced by the high expansion of F-7 relative to that of the EN-1. The experiment was repeated with a Kimble R-6 cladding which has a higher expansion than EN-1, and this proved successful.

Schott BaK-1 was drawn in a square rod using a Kimble EN-1 cladding. The rod was drawn without difficulty; however, excessive light loss appeared to be due to bubbles originating from small chips on the core corners. The experiment was repeated with the corners of the core bar fine ground to eliminate the chips, and an increase of 1% transmission was obtained. In a final effort a round core bar was used; however, no improvement resulted.

4.3 Cladding Glasses

Initial efforts in the investigation of new cladding glasses were to develop techniques for the use of fluorocrown glasses. The motivation is their very low index of refraction compared to conventional glasses. Their successful use would increase the numerical aperture obtainable in fibers of a given core glass. Where short wavelength transmission is important this is a more attractive answer than raising the index of refraction of the core glass.

Experiments were conducted with Schott FK-6, selected because of its very low index of refraction ($n_d = 1.446$). From both previous experience and experiments conducted under the project, the problem with these glasses was bound to be surface devitrification when heated in the atmosphere. Use of a low melting temperature frit to protect the surface was chosen as the most favorable approach without resorting to elaborate drawing modifications.

As an initial experiment for protecting the surface, a ground and polished bar of FK-6 was used as a core rod in a tube of Kimble KG-12. The intent was merely to study drawing characteristics and interface quality since the indices of refraction are not appropriate for an optical fiber. The results were poor. The KG-12 did not adhere well to the FK-6 and examination of the interface revealed devitrification.

In the next experiment a low melting temperature solder glass was used in an effort to obtain surface protection starting at a lower temperature. The FK-6 bar was coated with Corning 7570 frit in amyl acetate binder and drawn to 1/16" square. The four core surfaces represented four different conditions of surface roughness prior to coating. These varied from very rough to ground and polished. Examination of the fiber indicated that the polished surface had been quite well covered and protected.

Another bar of FK-6 with four ground and polished surfaces was coated with the 7570 frit and was drawn into 1/16" rods. These looked quite good at this stage. The 1/16" rods were then placed like barrel staves around a 1" square core of Schott SF-1 (n_d=1.72). This assembly was placed into a tube of Kimble KG-12 and drawn to about 1/16". This made a clad rod of .92 NA based on the indices

of the SF-1 and the FK-6. The KG-12 had no optical purpose. Its function was to facilitate drawing and to provide an outer cladding whose thermal expansion is low enough for adequate mechanical strength.

A 6x6 multifiber assembly was made from the SF1-FK6-KG12 rods and drawn to .0024". At this stage there was some transmission through the 10 micron elements, but it was poor compared to normal fibers.

The poor transmission indicated that some devitrification had occurred at a temperature below that at which the frit coating took place. An extensive effort was made to locate a frit glass with suitable properties; however, no glass meeting the requirements was available either commercially or experimentally. From the results of the experiments, however, success with fluorocrown glasses appears possible with an appropriate protective frit glass.

In addition to the fluorocrown experiments five cladding glasses were investigated as possible substitutes for Kimble R-6 and EN-1. The glasses, selected on the basis of their catalog properties as desirable optical grade claddings, are Schott BK-10, K-10, K-11, Ohara BK-7, and Pilkington Kovar sealing glass. Schott F-2 was used as a core material in each evaluation. Except for the Pilkington glass these glasses are unavailable in tubular form and the cladding was applied by the "barrel stave" technique.

Measurements made on rods fabricated with Schott BK-10 were encouraging and the glass was investigated further by attempting to draw multifiber. In this draw, however, excessive bubbles and devitrification resulted and the fiber obtained was lumpy and oversized. A second attempt was made with the mono rods prefused into a 1/4" square, but this fiber was also of poor quality again due to excessive bubbles and devitrification. From these experiments it appears that BK-10 is not able to withstand the required heat cycles and is therefore not a suitable cladding glass.

Somewhat better results were obtained with Schott K-10 and K-11 as cladding glasses and both types were drawn with Schott F-2 core glass into 10μ element multifiber ribbons using a prefusing operation. From each set of ribbons one fiberscope 1/8" x 1/8" x 40" was assembled. Transmission measurements on the multifiber and analysis of the fiberscopes, however, showed these glass combinations to be inferior to our standard in both transmission and blemish quality.

The remaining two glasses, Ohara BK-7 and the Pilkington Kovar glass showed a rod transmission lower than the other cladding glasses tried and therefore no further tests were performed.

Low transmission obtained in fibers made with experimental cladding glasses raised some question of the validity of substituting "barrel-stave" cladding for glasses unavailable in tubing form. As an experiment the "standard" Kimble R-6 cladding was also placed in "barrel staves" about an F-2 core. Transmission of the rods dropped substantially from that normally obtained using tubular R-6. In a second experiment the R-6 rods were cut in half and ground and polished to provide a cleaner interface. Again the transmission was substantially lower. In light of these results it would seem that several of the other glasses evaluated may in fact be suitable cladding candidates if they were made into tubular form and should be re-evaluated when such means become available.

4.4 Other Drawing Experiments

Most of the drawing experiments involved steps corresponding to those used in making multifiber fiberscopes. That is to say, they either led to the making of clad rod, or they involved an assembly of rods and were aimed at the making of multifiber ribbons. An exception was the making of 10 micron monofibers for the 3.6 meter light guide test described in Section 5.0. This test was an attempt to evaluate clad rods in a manner closely related to their intended use without complications that might be associated with the making of multifibers. For this test, clad rods were drawn to the 10 micron size in about the same furnace environment as would normally be used for drawing multifibers. Long light guides were made from the fibers and evaluated for the incidence of grey fibers.

Past experience in making 50 micron monofibers has indicated that the two step drawing procedure is less favorable for fiber quality than a single step draw. A test was devised to evaluate a single step versus a two step draw for making 10 micron fibers. This was done by making a rod and tube assembly one-fifth as large as that normally used for making 50 micron fibers and drawing directly to 10 microns without the intermediate clad rod step. This fiber was compared with fibers of the same materials drawn from clad rod and was found to be inferior to them. This indicated either that the clad rod step (which is fundamental to the making of multifibers) is relatively not harmful or that the compromises in scaling down the drawing of 50 micron fibers to 10 microns were relatively harmful. The glasses did receive an extra drawing step prior to cladding in making the one-fifth size set-up. There were also compromises in scaling the furnace to one-fifth size due to the increased problems of power input and insulation in a smaller heater. The assembly was run at one-fifth the input and output speeds used for 50 micron fibers, calculated to place the glasses at the same viscosities as for 50μ. The experiment may indicate that drawing speed is more significant to fiber quality than some other details of the drawing steps.

An experiment was performed to determine if poor quality of the Kimble R-6 tubing is a major cause of difficulty in drawing 10μ fiber for the 12-foot light guide test. R-6 tubing was drawn with no core to 3mm and then redrawn to 10μ. No difficulty was experienced in drawing the tubing. From this it appears that defects in the tubing are small enough and few enough so that the internal quality of the tubing itself is not the cause of failure. This result, however, does not preclude the possibility that defects present on the inner surface of the cladding might cause defects when a core is present.

5.0 Testing

5.1 Introduction

This section deals with the testing and evaluation of new materials and techniques explored under this contract. A series of tests was developed and employed for evaluating all the optical fibers and clad rod prepared during this investigation.

5.2 Glass Selection

In the investigation of suitable core glass candidates an initial selection of 11 glasses was made on the basis of past experience and/or favorable catalog data. Ten of these are Schott glasses. One glass, from Ohara, is approximately equivalent to Schott F-2 and the other remaining glass from Corning was a special run of flint glass. The main criterion for selection was bulk transmittance. An effort was made to select highly transparent glasses of relatively high index of refraction. The thermal expansion and transformation temperatures were also considered as indicators of their workability with cladding glasses.

The transmittance values given in the Schott catalog are for thicknesses of 5mm and 25mm. These were extrapolated to longer lengths, but were not considered sufficiently precise to predict the performance of long fibers. In an effort to obtain more reliable data, standard sized samples of the 11 glasses were procured and measured in the Cary and General Electric spectrophotometers. A sample length of 4" was selected, based on the limitations of the spectrophotometers. These were run with thin samples of the same materials in the reference beams in order to obtain internal transmittance curves for paths of 100mm. The curves obtained appeared to be more consistent than the extrapolated catalog values but were still not precise enough to give conclusive comparisons of peak transmittance among the best glasses. Three of the glasses (LaK-3, LaK-N12 and BaF-51) had significantly lower peak transmittance and were eliminated from further testing.

Table No. I lists the core glasses chosen and the measured peak transmittance values for each. Transmittances at 400nm are also given. Indices of refraction, thermal expansion, and transformation temperatures are included for reference. Eight of these glasses were selected for further evaluation and these are denoted by an asterisk. In addition to these, Schott BaK-1 and Corning 8378 were added later in the program.

TABLE NO. I

PROPERTIES OF CANDIDATE FIBER OPTIC CORE GLASSES

	Glass	Nd	α	T_G	T_{400}	T_{MAX}
	F-2 (Standard)	1.620	93	432°C	.93	.99
*	SF-4	1.755	90	420	.48	.99
*	SF-1	1.717	91	417	.65	.99
	LaK-3	1.694	90	610	.40	.93
	LaK-N12	1.678	90	621	.75	.96
	BaF-51	1.652	96	556	.67	.97
*	F-6	1.636	94	439	.89	.99
*	F-7	1.625	107	429	.81	.99
*	620-362 (Ohara)	1.620	99	---	.96	.99
*	BaF-4	1.606	88	540	.91	.99
*	LF-5	1.581	103	419	.98	.99
*	UK-50	1.523	85	554	.98	.99

α = Thermal expansion in/in X10^{-7}/°C 20°C-300°C

T_G = Transformation temperature

T_{400} = Internal transmittance over 100mm at λ = 400nm

T_{MAX} = Peak internal transmittance through 100mm

* indicates that the glass was also tested in longer lengths in fiber form

Six glasses were selected which showed properties favorable for an optical cladding. These glasses are listed in Table II with their manufacturer, index, expansion, and anneal point or transformation temperature. Properties of the "standard" glasses, Kimble R-6 and EN-1, are also given for reference.

5.3 Transmittance Measurements

The basic optical criterion for evaluation of the glasses tested under this program was transmittance. One way of expressing this property is by means of the absorption coefficient (K) defined by Lambert's law of absorption:

$$I = I_o e^{-Kx}$$

where I_o = the incident intensity

I = the transmitted intensity

x = thickness

K = absorption coefficient

The procedure used for the measurement of the absorption coefficient for fibers was to measure the intensity of radiation after it passes the intensity through a shorter length. Applying Lambert's law it is possible to calculate the absorption coefficient in the following manner:

$$I_1 = I_o e^{-Kx_1} \quad \text{for long length } x_1$$

$$I_2 = I_o e^{-Kx_2} \quad \text{for short length } x_2$$

solving for K:

$$K = - \frac{\ln(I_1/I_2)}{x_1 - x_2} = \frac{2.3 \log_{10}(I_2/I_1)}{x_1 - x_2}$$

where I_1 = intensity after passing through long length

I_2 = intensity after passing through short length

x_1 = length of long fiber

x_2 = length of short fiber

K = absorption coefficient

TABLE II

PROPERTIES OF CANDIDATE FIBER OPTIC CLADDING GLASSES

Manufacturer	Glass	N_d	α	A.P. or Tg
Kimble	R-6	1.52	93	525 (A.P.)
Kimble	EN-1	1.48	47	482 (A.P.)
Corning	7052	1.484	46	480 (A.P.)
Schott	K-10	1.502	74	459 (Tg)
Schott	K-11	1.501	70	493 (Tg)
Schott	BK-10	1.499	66	532 (Tg)
Ohara	BK-7	1.516	93	570 (Tg)
Pilkington	ME-1	1.487	49.5	515 (A.P.)

α = Thermal expansion in/in $X10^{-7}/^{o}C$ $20^{o}C$-$300^{o}C$

A.P. = Temperature for viscosity of 10^{13} poises

Tg = Transformation temperature (= A.P. approximately)

where x_1 and x_2 are in centimeters, K is expressed in terms of reciprocal centimeters. For convenience in discussion the attenuation was also tabulated in terms of loss per foot of length.

5.4 Apparatus

The apparatus used to measure the transmittance of fibers and clad rods consisted of an EG&G spectroradiometer system modified by incorporation of diffusing elements to provide diffuse input light and pickup diffuse output light. The system is shown schematically in Figure 35 and also in the photograph (Figure 36). The light source consists of a calibrated tungsten lamp with a well-regulated solid state power supply capable of maintaining lamp fluctuations to 0.25% rms. The lamp is housed in an especially designed enclosure which minimizes reflective errors and stray light.

The light is deflected for convenience in alignment by a pair of plane mirrors and strikes an opal glass diffuser. The sample was prepared by carefully scoring and breaking the end face to a reasonably flat and perpendicular surface. This was butted against the diffuser with a drop of immersion oil to further reduce the influence of the end face flatness. In the case of a clad rod the wall surface was covered with black plastic tape to avoid any reflection from the cladding-air interface. In the case of long flexible fibers this was impractical and deemed unnecessary.

The output end of the rod or fiber was placed against another opal glass diffuser and this was followed by still a third diffuser about three inches away and just ahead of the detector unit. This contained a photomultiplier and associated electronics. The readings were made on a sensitive meter capable of reading in the range from 10^{-11} to 10^{-6} amperes.

As the length of the rod or fiber was decreased the exit end was always returned to the same location ahead of the detector. In the case of the clad rods a number of readings was made at decreasing lengths and the results plotted as a function of length on semi-log paper. From this an average absorption coefficient, K, was calculated. This is the value given in Table No. III for each of the samples measured.

5.5 Results

Transmission measurements were made on over 50 experimental rods and fibers produced under this program. Results of all tests are given in Table No. III. This table is in three sections: 1) various core-cladding combinations, 2) various cladding glasses with F-2 core glass, and 3) various experimental techniques using F-2 core glass and three selected cladding glasses, mainly R-6. It can be seen that of the various core-cladding combinations the standard F-2, R-6 combination showed the lowest

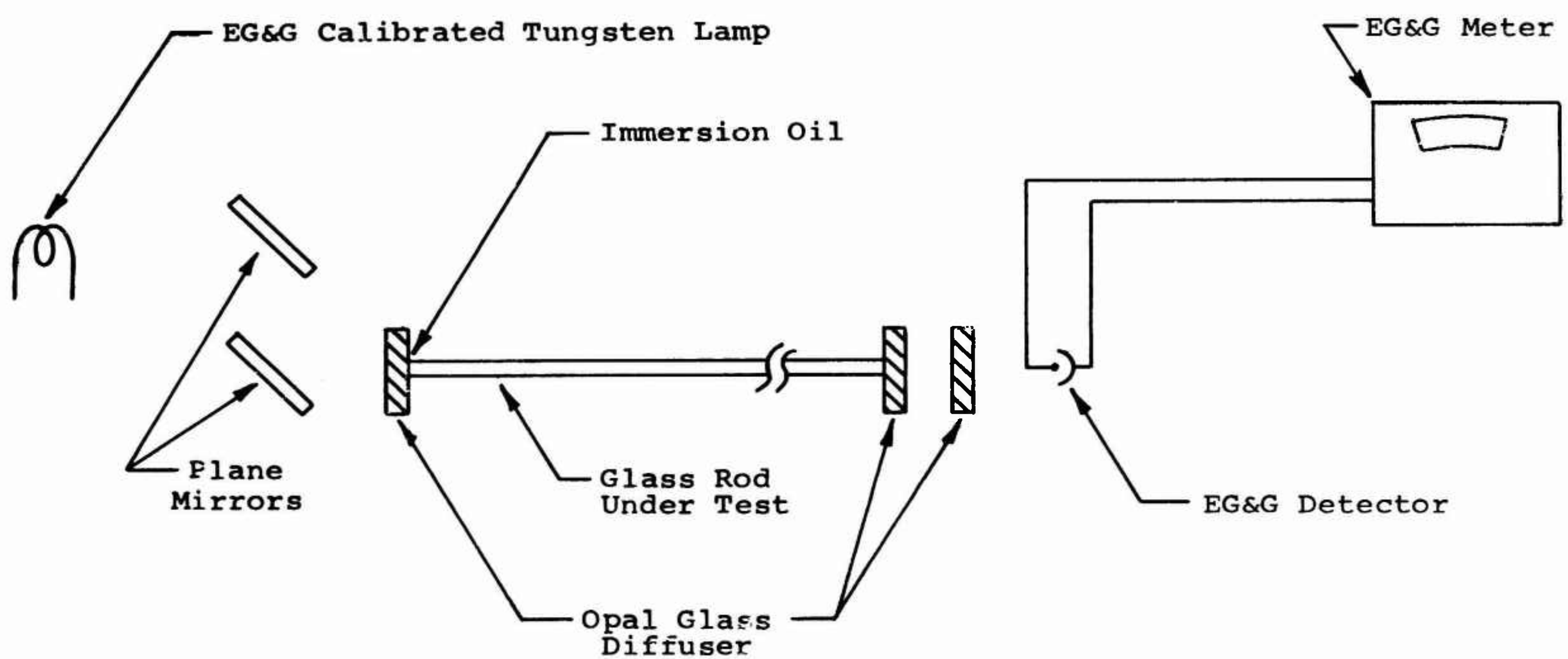

Figure 35 - Fiber Transmission Testing Equipment Schematic

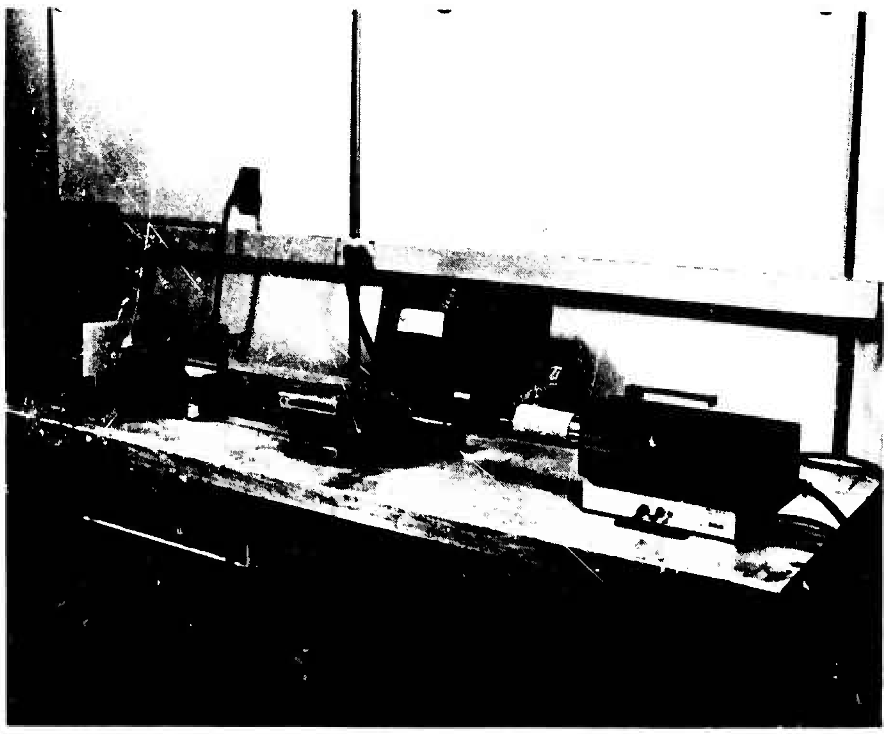

Figure 36 - Fiber Transmission Testing Equipment

TABLE NO. III - TRANSMISSION RESULTS ON EXPERIMENTAL FIBERS

SECTION 1 - EXPERIMENTAL CORE GLASSES

Core	Clad	Drawn	Tested	Type	Size	% Clad	K	Loss/Foot	Notes
F-2	R-6		6-25-69	Mono	.003"	25%	.0023/cm	6.9%	Light guide fiber from production.
F-2	R-6		6-27-69	Multi 10µ	65µ	25%	.0025	7.4%	Standard FS stock from production.
F-2	R-6		7-1-69	Round Rod	3mm	25%	.0022	6.5%	Std. clad rod stock from prod.
F-7	EN-1	10-28-69		Round Rod	3mm	30%			EN-1 too low expansion for F-7. Fiber breakage occurred.
F-7	R-6	10-30-69	11-3-69	Square Rod	2x2mm	31%	.0086	23%	
620-362 (Ohara)	7052	10-29-69	11-3-69	Round Rod	3mm	36%	.0042	12%	
SF-1 (Square Core)	EN-1	10-17-69	10-27-69	Round Rod	3mm	35%			Did not transmit. Cladding peeled off. Core too soft.
UK-50	EN-1	10-1-69	10-7-69	Round Rod	3mm	31%	.0036	10%	
LF-5	7052	10-14-69	10-16-69	Round Rod	3mm	33%	.0041	12%	
BaF-4	7052	10-14-69	10-16-69	Round Rod	3mm	34%	.0041	12%	

Table No. III (Cont'd)

Core	Clad	Drawn	Tested	Type	Size	% Clad	K	Loss/Foot	Notes
F-6	7052	10-14-69	10-27-69	Round Rod	3mm	36%	.0069/cm	19%	
SF-4 (Square Bar)	EN-1	10-17-69	10-20-69	Round Rod	3mm	37%			Did not transmit due to excessive bubbles
SF-4	R-6	10-29-69	11-3-69	Round Rod	3mm	36%	.0077	21%	
SF-4	EN-1	12-9-69	12-9-69	Round Rod	3mm	37%	.009	24%	
BaK-1	EN-1	10-28-69	10-30-69	Square Rod	3x3mm	35%	.0081	22%	Excessive light loss through corners due to fracturing of core glass
BaK-1	EN-1	11-17-69	11-21-69	Square Rod	3x3mm	23%	.0076	21%	Corners fine ground
BaK-1	7052	12-5-69	12-9-69	Round Rod	3mm	34%	.0104	28%	
BaK-1	7052		12-10-69	Round Rod	3mm	34%	.0098	26%	Retest of rod drawn 12-5-69
BaK-1	EN-1		12-10-69	Square Rod	3x3mm	23%	.0072	20%	Retest of rod drawn 11-17-69
8378 (Corning)	R-6	4-6-70	4-13-70	Round Rod	3mm	35.5%	.0050	14%	Special run of core glass

Table No. III (Cont'd)

SECTION 2 - EXPERIMENTAL CLADDING GLASSES

Core	Clad	Drawn	Tested	Type	Size	% Clad	K	Loss/Foot	Notes
F-2	7052	10-14-69	10-16-69	Round Rod	3mm	32%	.002/cm	6.0%	
F-2	BK-10	11-12-69	11-13-69	Round Rod	3mm	20%	.0031	9.0%	Strip cladding
F-2	BK-10	11-17-69		Multi 10μ		20%			Attempt to draw .0024" multifiber in drusing fixture unsuccessful. Excessive devitrification & bubbles caused lumped fiber. No transmittance tests.
F-2	BK-10	11-26-69		Multi 10μ	.0024"	20%			Mono rods prefused to 1/4' square rod. Fiber was poor due to excessive devitrification & bubbles. No transmittance tests.
F-2	K-11	12-5-69	12-9-69	Round Rod	3mm	20%	.0031	9.0%	Strip cladding
F-2	K-11	12-8-69	12-11-69	Multi 10μ	.002	20%	.0136	34%	Prefused multi rods. One FS assembled 1/8"x1/8"x40". Excessive absorption.
F-2	K-10	9-24-69	9-30-69	Round Rod	3mm	20%	.0053	15%	Strip cladding
F-2	K-10	9-29-69	12-18-69	Multi 10μ	.0025"	20%	.0059	17%	FS assembled 10-3-69

Table No. III (Cont'd)

Core	Clad	Drawn	Tested	Type	Size	% Clad	K	Loss/Foot	Notes
F-2	BK-7	1-12-70	1-26-70	Round Rod	3mm		.005/cm	14%	Round strip cladding
F-2	Pilkington	4-6-70	4-13-70	Round Rod	3mm	42.5%	.0042	12%	
F-2	R-6	6-10-69	9-12-69	Round Rod	3mm		.0112	29%	Double crucible
F-2	7052	11-5-69	12-9-69	Round Rod	3mm	36%	.0034	11%	5g 7570 frit (puddled). Poor quality--bubbles at interface
F-2	EN-1	12-5-69	12-8-69	Round Rod	3mm	16%	.0044	14%	FL-3 interface drawn with production facilities
F-2	R-6	1-26-70	1-30-70	Round Rod	3mm		.004	12%	Induced heat experi. Best of lot
F-2	EN-1	1-5-70		Round Rod	3mm				7570 fritted interface, poor quality, excess. bubbles, white color, no testing performed
F-2	R-6	2-16-70	2-20-70	Round Rod	3mm	37%	.006	18%	Round strip cladding Purpose to evaluate strip vs tube clad
F-2	R-6	2-15-70	2-18-70	Round Rod	3mm	33.7%	.003	8.0%	Acid rinsed core & clad to remove hydrated layer.

Table No. III (Cont'd)

Core	Clad	Drawn	Tested	Type	Size	% Clad	K	Loss/Foot	Notes
F-2	R-6	3-9-70	3-11-70	Round Rod	3mm	36%	.0016/cm	5.2%	Production facility sample after change to 15" vacuum. Rod looked very good.
F-2	R-6	3-9-70	3-13-70	Round Rod	3mm	36%	.0044	13%	Production sample with 15" vacuum. Rod looked very bad.
F-2	R-6	3-16-70	3-16-70	Round Rod	3mm	36%	.0026	7.9%	Production sample with 15" vacuum. Rod looked very avg.
F-2	R-6	3-2-70	3-11-70	Round Rod	3mm	36%	.0044	11.5%	Vacuum baked assy.
F-2	R-6	3-9-70	3-16-70	Round Rod	3mm	36%	.0032	9.2%	Differential draw with 6" preheater
F-2	R-6	3-16-70	3-18-70	Round Rod	3mm	36%	.0018	5.6%	Best sample of exper- imentally drawn rods using prod. facili- ties with 20" vacuum
F-2	R-6	3-9-70	3-18-70	Round Rod	3mm	36%	.0026	7.9%	Best sample of experimentally drawn rods using production facilities with no vacuum
F-2	R-6	3-9-70	3-18-70	Round Rod	3mm	25%	.0031	9.0%	Differential draw. No preheat.
F-2	EN-1	3-16-70	3-18-70	Round Rod	3mm	25%	.0038	11%	Differential draw. No preheat.
F-2	R-6	3-16-70	3-20-70	Round Rod	3mm		.0034	10%	Press fused mono assembly

Table No. III (Cont'd)

Core	Clad	Drawn	Tested	Type	Size	% Clad	K	Loss/Foot	Notes
F-2	R-6	3-16-70	3-26-70	Round Rod	3mm	35%	.002/cm	6.2%	Steel ring heat sink around bundle. Full vacuum
F-2	R-6	3-16-70	3-26-70	Round Rod	3mm	35%	.002	6.0%	Same run as above. Vacuum reduced to 1/2
F-2	R-6	3-16-70	3-26-70	Round Rod	3mm	40%	.0042	12%	Double crucible 3/8" rod redrawn with std. clamp machine. Many bubbles observable.
F-2	R-6	3-23-70	3-26-70	Round Rod	3mm	36%	.002	6.1%	Random production sample drawn with 15" vacuum
F-2	R-6	3-26-70	3-30-70	Round Rod	4mm	35%	.0049	14%	Double crucible after addition of bottom heater. Transient conditions
F-2	R-6	3-23-70	3-30-70	Round Rod	3mm	24%	.0094	25%	R-6 clad in form of semi-cricular strips
F-2	R-6	3-23-70	3-30-70	Round Rod	3mm	25%	.0028	8.5%	Differential draw. 3 furnaces
F-2	R-6	4-6-70	4-10-70	Round Rod	3mm	36%	.0023	6.8%	Reject production rod. Diffused interface
F-2	R-6	4-6-70	4-13-70	Round Rod	3mm	36%	.0022	6.5%	Avg. production rod. Diffused interface.
F-2	R-6	4-6-70	4-13-70	Round Rod	3mm	36%	.0034	9.8%	Predrawn core and clad
F-2	R-6	4-13-70	4-15-70	Mono-fiber	.003	36%	.0057	16.0%	Drawn from diffused interface rod of average quality

Table No. III (Cont'd)

Core	Clad	Drawn	Tested	Type	Size	% Clad	K	Loss/Foot	Notes
F-2	R-6	4-20-70	4-23-70	Round Rod	3mm	36%	.0024/cm	7.0%	Reject production rod. Diffused interface
F-2	R-6	4-20-70	4-24-70	Round Rod	3mm	36%	.0034	10.0%	Reject production rod.
F-2	R-6	4-27-70	4-28-70	Round Rod	3mm	36%	.0051	9.5%	Std. production rod. #29 Wratten filter (610 mμ+) input
F-2	R-6	4-27-70	4-29-70	Round Rod	3mm	36%	.0012	4.2%	Std. production rod. 550 mμ input

attenuation. On this basis it was decided to select F-2 as the reference core glass to evaluate a number of cladding glasses.

The results of this series of tests is shown in the second section of the table. It was found that only one cladding glass, Corning Type 7052, gave a better result than the standard R-6 cladding glass; however, the difference was small and probably within the accuracy of the measurement. It is likely that some of the other cladding glasses would have performed better if they had been available in tubing form; however, there was no apparent advantage in using them so that R-6 which is more easily workable than 7052 was selected for most of the experimental drawing techniques described in the previous sections and tabulated in the third section of Table No. III.

5.6 Additional Tests

In addition to the transmittance tests described in the previous section three other tests were performed in an effort to derive additional information about the losses in the experimental rods and fibers produced in this investigation.

One of these tests consisted of drawing the experimental clad rods into small monofibers to determine whether the attenuation found in the rods could be correlated with a random loss in transmission in the resulting fibers. In the multifibers normally drawn for image-transmitting fiberscopes the final fiber size is 10 microns so this was the size fiber into which the clad rods were drawn.

The test consisted of preparing bundles of a known number of these small monofibers 3.6 meters long and counting the number of defective fibers to determine the percentage contained. Two sets of bundles were prepared in this manner in an effort to evaluate the influence of one of the process variations. The first two bundles consisted of 10,000 fibers each, one made from the F-2, R-6 glass combination and the standard cleaning process and the other from the same glasses but acid cleaned to reduce surface hydration. The second set of bundles were made in the same manner but consisted of only 1,000 fibers each to reduce the counting problem. The results were as shown in Table No. IV.

TABLE NO. IV

Sample	Total Fibers	% Defective
Standard Cleaning	10,000	5.4%
Acid Treated	10,000	8.2%
Standard Cleaning	1,000	6.5%
Acid Treated	1,000	6.0%

It can be seen from these results that this test was unable to distinguish between the two experiments. The principal difficulty was in determining when a fiber was significantly lower in transmission by visual inspection alone. Thus the test proved to be mainly a qualitative one and was not pursued for other glass combinations or experimental runs.

A study was made to determine the residual losses in a standard F2/R6 rod. Spectal intensity measurements made on the transmission test system plus spectrophotometric absorption measurements made on an 8" sample of bulk Schott F-2 glass revealed that the losses in a fiber attributable to the interface are significantly less than the absorption loss in the core glass. The spectrally integrated absorption of an F-2 core rod was determined to be 5.4% per foot. Subtracting this from the total loss of 6.5% per foot in a good F2/R6 clad rod indicates that the interface loss is only 1.1% per foot.

The above results were confirmed by making transmission measurements on F2/R6 rods with a limited spectral input using the EG&G apparatus. At an input of 550mμ, where the F-2 glass absorption is a minimum, a standard rod measured a loss per foot of only 4.2%. On the other hand, when the input was limited to the red region of the spectrum where the F-2 glass has a minor absorption band the transmission of a standard rod showed an increased loss per foot of 9.5%.

Another relevant observation regarding the relative importance of core glass absorption versus interface losses was the relatively small decrease in transmission as the fiber diameter was decreased. Theoretically, there is some fixed amount of loss associated with each reflection of a particular ray within the fiber. This may be due to absorption in the cladding glass or scattering from the interface defects. The number of reflections, N, occurring within a fiber of diameter, d, and length, L, is given by

$$N = \frac{L}{d} \frac{\tan \theta}{\cos \phi}$$

where θ and ϕ define the orientation of a ray within the fiber. In the absence of absorption losses the transmission, T, in traversing the fiber will then be given by:

$$T = R^N$$

where R is the reflectivity at each reflection of the ray. In making fiber transmission measurements the angular distribution of the input rays is assumed to remain invarient, therefore, it follows that

$$T = R^{\frac{LC}{d}}$$

where $C = \frac{\tan \theta}{\cos \phi}$ = constant

Measurements made on standard F2/R6 fibers revealed the following transmission fall off. The data was obtained by the transmission test described earlier and is an average of five trials for each fiber size.

Fiber Diameter	Transmission/Foot
.120"	93.5%
.003"	93.1%
.00043"	92.6%

The diameters given above correspond to the total fiber diameter. The cladding fraction used for these rods was 25% and we can use that to obtain the diameter of the core which is the parameter of interest.

Subtracting the absorption loss in the core of 5.4%/foot we have:

Fiber Diameter	Core Diameter	Transmission/Foot
.120"	.104"	98.9%
.003"	.0026"	98.5%
.00043"	.00037"	98.0%

Using this data a calculation was made for the value of R. This was determined to be in excess of .99999/reflection.

Thus the loss due to core glass absorption is the major cause of attenuation in fibers and particularly in clad rods where it accounts for more than 80% of the total loss. In retrospect, a greater emphasis on core glass improvement would have been appropriate in view of the relative contributions of absorption and interface scattering to the overall attenuation.

6.0 Summary

The two principal goals of this contract were: 1) to investigate materials and methods for improving the transmission of optical fibers, and 2) to develop a method for fusing the ends of coherent fiber bundles (fiberscopes) to improve their image quality.

The transmission efficiency of optical fibers depends on two main factors, the transmittance of the bulk core glass and the quality of the reflecting interface between core and cladding. For this investigation it was decided to limit the choice of core glasses to available cataloged glasses rather than attempt to develop entirely new glasses with higher transparency. The principal source for these glasses was the well-known firm of Schott which has the widest selection of cataloged glasses and from experience was known to produce many glasses of high transmittance. A number of promising candidates were selected and samples procured. These were measured for transmittance in 100mm lengths and the best selected for drawing into fibers.

Similarly the cladding glasses were chosen from among commercially available optical and technical glasses and were selected for their thermal properties and refractive indices. Promising combinations of core and cladding glasses were drawn into fibers and clad rods and tested for transmittance. Based on these measurements it was found that the most reliable high transmission combination was the one already commonly used, namely Schott F-2 for the core glass and Kimble R-6 for the cladding glass. This combination was chosen for most of the experiments designed to improve the interface quality. Kimble EN-1 and the equivalent Corning 7052 were also used as cladding glasses in these experiments.

Two main areas for interface improvement were explored: 1) the use of fluxes to minimize interface defects between the core rod and the cladding tube, and 2) the use of a double platinum crucible to remelt the core and cladding glasses and thereby provide a pristine core-clad interface. Although certain of the fluxing experiments showed some promise of controlling the bubble formation at the interface, the resulting clad rods had increased rather than reduced attenuation. It was concluded that the flux often introduced more light scattering defects than it eliminated.

The experiments on the double platinum crucible were hampered by difficulties in providing a suitable heater and several different approaches were tried in order to obtain the required viscosity control. Only a few successful runs of clad rod were obtained and these did not show the hoped-for improvement

in transmission. We are still convinced, however, that this approach is fundamentally correct and that significantly improved interface quality can be achieved by this route.

A number of variations of the standard rod and tube drawing process were also investigated including special preparation of the core and cladding glass surfaces, pre-baking, controlling the contact angle of the core and cladding glasses during fusion and varying the vacuum between the core and cladding. Most of these resulted in increased attenuation. One approach which provided some improvement involved placing a metal ring heat sink into the upper part of the drawing furnace. This had the effect of delaying the core-clad fusion until more outgassing could take place and increased the contact angle to reduce gas trapping. Even so, the resulting rods were only slightly better than the average from the conventional rod and tube production process.

The experiments on fusing coherent multi-fiber bundles proved to be quite successful in bundles of 3x3mm in cross section. The objectionable grid pattern and interstitial voids characteristic of the conventional cemented bundle were significantly improved by the fusing process as had been predicted. When the process was extended to larger bundles (10x10mm cross section) fiber dislocation became a problem and overshadowed the improvement obtained by fusing the fibers together. It was felt, however, that with additional experimentation this process could be successfully extended to 10x10mm bundles or even somewhat larger if required.

Unclassified

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) American Optical Corporation 14 Mechanic Street Southbridge, Massachusetts 01550	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP

3. REPORT TITLE

Exploratory Development of Improved Optical Fiber Bundles

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Final Report - 1 March 1969 to 30 June 1970

5. AUTHOR(S) (First name, middle initial, last name)

Ethan C. Hopkins
Walter P. Siegmund and Henry B. Cole

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
January 1971	70	None

8a. CONTRACT OR GRANT NO. F33615-69-C-1391	9a. ORIGINATOR'S REPORT NUMBER(S)
b. PROJECT NO.	
c.	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)
d.	None

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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
None	Air Force Materials Laboratory Air Force Systems Command Wright-Patterson Air Force Base, Ohio 45433

13. ABSTRACT

The purpose of this contract was to conduct exploratory development on materials and techniques to improve glass optical fibers and fiber bundles with particular reference to coherent multifiber fiberscopes. Studies were carried out on improved interface formation, end tip fusing, experimental fiber drawing techniques and evaluation of various component glass, clad rods and fibers. Two basically different approaches to improving the core-cladding interface were explored: 1) the use of low melting fluxes with the rod and tube method, and 2) the use of a double crucible to completely melt the core and cladding glasses before drawing. Neither approach provided significantly better interface quality in the resulting clad rods.

A method was developed for fusing the end tips of coherent fiberscope bundles. Good results were achieved in bundles up to 3x3mm in cross section but considerable difficulty was encountered when the bundle size was increased to 10x10mm.

A number of different glass combinations were selected and drawn into clad rods and fibers in a search for improved transmission efficiency. It was concluded that the properties of commercially available glasses presently limit the fiber transmission and that glasses with improved bulk transmittance must be formulated if improved fibers are to be realized.

DD FORM 1 NOV 65 1473

Unclassified

Security Classification